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(21) International Application Number: PCT/CA00/00137 (22) International Filing Date: 15 February 2000 (15.02.00) (30) Priority Data: 60/120,971 19 February 1999 (19.02.99) US (71) Applicant: HER MAJESTY IN RIGHT OF CANADA as represented by THE MINISTER OF NATIONAL DEFENCE [CA/CA]; Department of National Defence, 101 Colonel By Drive, Ottawa, Ontario K1A 0K2 (CA). (72) Inventors: PURDON, J., Garfield; 12 Calder Green S.E., Medicine Hat, Alberta T1B 3K6 (CA). CHENIER, Claude, L.; 9 Hewitt Court N.E., Medicine Hat, Alberta T1C 1N8 (CA). BURCZYK, Andrew, F., H.; P.O. Box 1361, 17-5th Street, N.E., Redcliff, Alberta T0J 2P0 (CA). (74) Agent: GOODWIN, Sean, W.; The Law Office of Sean W. Goodwin, Suite 300, 714 First Street S.E., Calgary, Alberta T2G 2G8 (CA).		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: BROAD SPECTRUM DECONTAMINATION FORMULATION AND METHOD OF USE		
(57) Abstract <p>A decontamination formulation is provided which is effective against a broad spectrum of chemical and biological warfare agents and radioactive dusts, comprising an active decontamination agent, a co-solvent, a buffer system to optimize the initial reaction pH above 8.5 and more preferably in the range of 10 to 11 for favoring oxidation of VX and HD and hydrolysis of G agents, and a surfactant similar to fire-fighting foaming agent. Formulations comprise, in water by weight, 1% to 15% of a hydrated chloroisocyanuric acid salt, 1% to 10% of a PPG co-solvent, 1% to 15% surfactant and a buffer system to initially maintain said formulation at a pH from about 11 to about 8.5 for sufficient duration to effect decontamination. The formulation can be provided in kit form or concentrate form, be prepared, in part, in advance or on site, and be dispensed in foam form which aids in coating and adhering of the decontamination formulation to contaminated surfaces. All ingredients can be pumped through a foam nozzle or water, co-solvent and surfactant can be pumped to the nozzle with solutions of buffer and of active ingredient being introduced at the nozzle for minimizing pump exposure.</p>		

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1 **"BROAD SPECTRUM DECONTAMINATION FORMULATION**
2 **AND METHOD OF USE"**

3
4 **FIELD OF THE INVENTION**

5 The present invention relates to decontamination formulations and
6 more particularly to formulations for decontaminating surfaces and/or materials
7 contaminated with chemical and/or biological warfare agents and/or nuclear
8 radioactive particles.

9
10 **BACKGROUND OF THE INVENTION**

11 Chemical (CW) and biological (BW) warfare agents (collectively CB
12 agents) are becoming an increasingly important part of defence weaponry.
13 Further, radioactive fallout or dusts have also been of concern, since nuclear
14 devices have been added to military arsenals.

15
16 **Nuclear/Radioactive Particles**

17 Nuclear or radioactive particles pose a significant threat to
18 personnel due to the effects of ionizing radiation. In addition to the initial release
19 of radiation from a nuclear device and radiation caused by emission from
20 materials which have become radioactive as a result of the initial detonation,
21 inhalation of radioactive dusts or particulate matter can lead to significant
22 numbers of casualties long after the attack. As with BW agents, secondary
23 aerosolization poses an ever-present threat and results in the need to wear
24 protective masks for extended periods of time.

25

1 Biological Warfare (BW) Agents

2 BW agents are characterized as microorganisms including bacteria,
 3 viruses and fungi. They are particulate in nature and present a significant hazard
 4 long after an attack through formation of secondary aerosols which are inhaled.
 5 Unlike CW, BW agents may not result in immediate effects. A lapse of hours,
 6 days or weeks may occur before the full extent of their effects become apparent.
 7 In the case of certain BW agents, like anthrax, spore production ensures that the
 8 BW agent can remain in the environment for years while retaining biological
 9 activity. While BW agents may be readily removed from a surface they are often
 10 merely repositioned in the underlying environment and remain hazardous if
 11 disturbed.

13 Chemical Warfare (CW) agents

14 Three main types of persistent and semi-persistent CW agents
 15 exist. They are vesicants and two families of nerve gases, V and G, as outlined in
 16 Table I.

17 Table I

18	Family	Agent	Acronym	Formula
19	Vesicants	Sulfur Mustard	HD	$\text{Cl-CH}_2\text{CH}_2\text{S-CH}_2\text{CH}_2\text{Cl}$
20		Nitrogen Mustard	HN-1	$(\text{CH}_2\text{CH}_2\text{Cl})_2\text{NC}_2\text{H}_5$
21		Nitrogen Mustard	HN-2	$(\text{CH}_2\text{CH}_2\text{Cl})_2\text{NCH}_3$
22		Nitrogen Mustard	HN-3	$\text{N}(\text{CH}_2\text{CH}_2\text{Cl})_3$
23		Lewisite	L	ClCH=CHAsCl_2
24	G	Tabun	GA	$\text{C}_5\text{H}_{11}\text{N}_2\text{O}_2\text{P}$
25		Sarin	GB	$\text{C}_4\text{H}_{10}\text{FO}_2\text{P}$
26		Soman	GD	$\text{C}_7\text{H}_{16}\text{FO}_2\text{P}$
27	V		VX	$\text{CH}_3\text{CH}_2\text{O-P(O)(CH}_3\text{)-SCH}_2\text{CH}_2\text{N(C}_3\text{H}_7\text{)}_2$

28
 29 Vesicants act as blistering agents that attack skin and mucous
 30 membranes and are lethal at high doses.

31 The V agents are in the phosphorylthiocholine class of compounds,
 32 while the G agents are phosphonofluoridates. Both share the same reaction

1 chemistry as organophosphorous esters and pesticides. Nerve agents act on the
2 central nervous system by reacting with the enzyme acetylcholinesterase to
3 cause respiratory collapse, convulsions and death.

4 G-agents tend to be semi-volatile and toxic by inhalation and
5 percutaneous absorption, while V-agents are relatively non-volatile, persistent,
6 and very toxic by the percutaneous route.

7 The threat of the use of CB agents and nuclear devices has
8 prompted the need to develop protective and decontamination measures for
9 personnel and military hardware.

10

11 Decontamination - Radioactive Particles

12 As radioactive particles are nuclear in origin, decontamination cannot
13 deactivate the radioactive hazard. However, the removal of the particulate matter
14 from equipment can significantly reduce aerosolization potential and the spread
15 of the radioactive hazard to clean areas. Generally, removal of the particulate
16 matter requires the encapsulation of the particles and subsequent removal of the
17 encapsulated material from equipment surfaces.

18 Decontamination - BW Agents

19 In the case of BW agents, personal protective equipment such as
20 masks, protective suits etc. are the primary defence against contamination. In
21 some cases, where time and environmental conditions exist, natural weathering
22 such as exposure to sunshine, heat and moisture may destroy the BW agent.

23 For many BW agents, standard disinfectants can be very effective
24 as decontaminants. An example is the use of hypochlorites or chlorine gas in the
25 treatment of water supplies, swimming pools and in sanitizing food preparation

1 equipment. Active chlorine is considered to be among the most economical yet
2 most effective broad spectrum BW agent decontaminant. Hypochlorites have
3 been shown to be effective against some of the most robust BW agents such as
4 anthrax spores as well as viruses and bacteria. Hypochlorous acid is superior to
5 that of hypochlorite anion as it more readily crosses the cell membrane. Thus, it
6 would be advantageous to perform decontamination of BW agents in a slightly
7 acidic, neutral or slightly basic media where hypochlorous acid is a dominant
8 active component rather than in a strongly basic solution, where hypochlorite
9 anion is the predominant species.

10

11 Decontamination - CW Agents

12 CW agent decontamination presents a number of challenges.
13 Following a CW agent attack, the semi-persistent or persistent nature of these
14 agents allows them to remain toxic, not only during dissemination, but also for
15 many hours or even days after the attack. The principal hazard occurs through
16 direct inhalation of the vapor off-gassed from the agent or through physical
17 contact with the skin or mucous membranes, through which it is absorbed.

18

19 Generally

20 Ideally, a decontamination formulation should be broad-spectrum in
21 nature, as in most cases the actual nature of the warfare agents being faced is
22 not known. It should be compatible with, and non-corrosive to, equipment used in
23 its application as well as to the equipment to be decontaminated. It should not
24 soften nor damage paints, coatings, polymeric seals or gaskets or transparencies
25 such as windscreens. It should not interfere with in-service monitoring equipment

1 used to verify the effectiveness of the decontamination or to locate residual
2 contamination. It should be easy to prepare, easy to apply and remove, and
3 remain stable for reasonable lengths of time after preparation. It is highly
4 desirable that it adhere to and coat vertical surfaces for sufficient periods of time
5 for agent desorption from the surface and detoxification, yet be easy to remove
6 by evaporation or by rinsing. If used in combination with a surfactant, the
7 decontamination formulation should not compromise the integrity of the foam. It
8 should be of low toxicity, be non-flammable and have a low impact on the
9 environment in order that training can be realistically and frequently performed.
10 Preferably, the formulation should be based in media capable of solubilizing and
11 supporting detoxification of the sparingly soluble CW agents and solubilizing and
12 degrading polymeric thickeners in which the CW agent may reside. Often, these
13 thickeners have high adherence to surfaces and are more difficult to remove than
14 the agents in neat form. Where possible, the decontaminant should be in a
15 concentrated form for mixing with water or other suitable diluent in order to
16 reduce logistical loads on transport and storage and should be readily mixed. For
17 economic reasons it should be formulated from compounds that are readily
18 available in large quantities and be stable in storage for long periods of time.
19 Ideally, the media for dilution should be water or seawater as, in most cases, it is
20 readily available on site and is non-toxic.

21 Prior art decontamination formulations have taken advantage of the
22 fact that CW agents can generally be oxidized or hydrolyzed, dependent upon
23 their structure, to result in non-toxic products. Many BW agents are readily
24 decontaminated by those same active ingredients, such as hypochlorite and
25 radioactive particles are encapsulated by the surfactants utilized to cause the

1 formulations to adhere to vertical surfaces and are removed and diluted during
2 the removal of the formulation, generally by washing.

3 In the case of V agents, mustards and biological warfare agents,
4 oxidation has been most successful. Various reactants such as hypochlorites,
5 permanganates, N-chloro and N-bromo compounds, ozonizing compounds and
6 peroxides have been used.

7 G-agents are not easily oxidized, therefore hydrolysis is normally
8 utilized to address this family of agents. Although hydrolysis can be effective with
9 mustards, they must be in solution before they can be hydrolyzed. Hydrolysis can
10 be accomplished using hydroxides or hypochlorites acting as catalyst, and by
11 water, often with the addition of metal salts to catalyze the reaction. Hydrolysis
12 utilizing enzymes such as organophosphorus acid anhydrase has been studied,
13 although large scale broad spectrum decontaminants are not yet available using
14 this approach.

15 Nucleophilic displacement can be used to decontaminate nerve and
16 vesicant agents. Since it involves replacement of one group with another less
17 active one, the processes of oxidation and hydrolysis are not necessarily
18 employed. In order to be effective, a formulation utilizing nucleophilic
19 displacement must provide stoichiometric replacement species for all of the CW
20 agents it may encounter, thus adding to the logistical load of transport and
21 storage.

22 Among the first decontaminants to be used was bleach powder and,
23 to a much lesser degree, potassium permanganate. Bleach can convert CW
24 agents into inert products at the liquid (Bleach solution) or liquid-solid (bleach
25 powder) interface within a few minutes via vigorous oxidation and elimination

1 reactions. However, there are disadvantages. The active chlorine content in
2 bleach decreases gradually with storage time, hence an excess amount of bleach
3 is needed for the oxidation of some agents. In addition, its alkalinity can be
4 corrosive to metal surfaces. Its effectiveness is limited to removing agents from
5 surfaces, since it is not effective in removing agents that have already penetrated
6 into paints.

7 Following the use of bleach as a decontaminant, the US Army
8 introduced Decontamination Solution 2 (DS2), which is a wide-spectrum, ready-
9 to-use, chemically reactive nucleophilic decontaminant, having long-term stability
10 over an extended range of temperature of -26°C to 52°C. This polar non-aqueous
11 liquid consists, by weight, of 70% diethylenetriamine, 28% ethylene glycol
12 monomethyl ether and 2% sodium hydroxide. At ambient temperature, it reacts
13 with any of the HD, VX, GB or GB agents within a few seconds. Typically, DS2 is
14 premixed and stored in 1.3qt cans, 5-gallon pails and 14-L containers.

15 However, DS2 does have drawbacks. It is a highly aggressive
16 chemical solution that is toxic and flammable. It damages paint, plastics, rubber
17 and leather materials and, in use, leads to rapid corrosion and oxidation of some
18 metals. It must be used in its premixed form, which poses a logistical transport
19 problem. DS2 is corrosive to the skin, requiring personnel handling it to wear
20 respirators with eye shields and chemically protective gloves to avoid skin
21 contact. Ethylene glycol monomethyl ether has been identified as being toxic to
22 personnel.

23 Another popular decontaminant is the German Emulsion (C8)
24 system. This system consists, by weight, of 76% water, 15% perchloroethylene,
25 1% anionic surfactant and 8% high-test-hypochlorite (HTH). Many of the benefits

1 of this system are attributed to the perchloroethylene continuous phase. C8 is of
2 low corrosivity despite the high pH of the aqueous phase. It is effective in
3 dissolving thickeners and can penetrate paint and react with the embedded
4 agents, without damaging the paint. It is viscous enough to provide a thin and
5 coherent film on the surface to allow sufficient time for reaction with the agents.

6 C8 has several drawbacks. It must be mixed for periods of up to an
7 hour prior to use to generate the emulsion. Even then, it is possible that no
8 emulsion will form. Perchloroethylene has recently been identified by the
9 Canadian and other governments as being environmentally unacceptable and its
10 production and use has been discouraged. The eventual goal is to completely
11 phase out its production. Removal of the perchloroethylene from the
12 decontaminant would render it incapable of solubilizing thickened agents and
13 dissolving highly insoluble CW agents. The surfactant designed to form the
14 emulsion is difficult to obtain. It was originally only available from a manufacturer
15 in West Germany, which has recently discontinued its production.

16 Clearly, given the drawbacks of the existing decontamination
17 formulations, it is necessary to develop a formulation that is stable, non-toxic to
18 personnel and to the environment, of low corrosivity, effective against a broad
19 spectrum of CW agents, BW agents, and, optionally, radioactive particulate
20 matter, prepared on site in a substantially aqueous medium and capable of
21 coating surfaces, including vertical surfaces, for a minimum of 30 minutes as
22 outlined by NATO.

23 It is clear that the effectiveness of any formulation does not rely
24 solely on the active ingredient, but rather with its overall composition.

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1

SUMMARY OF THE INVENTION

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A decontamination formulation is provided which is effective against a broad spectrum of chemical and biological warfare agents, including those with persistent spore production. Further, it is capable of encapsulating particulate radioactive material for facilitating efficient removal by scrubbing and/or rinsing.

In a simplified aspect of the present invention, the decontamination formulation comprises a synergistic combination of an active decontamination agent, a co-solvent preferably undetectable by decontamination monitoring equipment which aids in solubilization of relatively insoluble chemical warfare agents and thickened agents, a buffer system to optimize the initial reaction pH above 8.5 and more preferably in the range of 10 to 11 for favoring oxidation of VX and HD and hydrolysis of G agents, and finally a surfactant to aid in encapsulation of particulate matter and formation of a reliable foam of uniform bubble size when aerated. The surfactant enables foaming of the formulation for coating of surfaces including adherence to vertical surfaces. This coating is stable for sufficient time to ensure effective contact and decontamination. The formulation of the present invention is soluble in an aqueous medium and the use of gray or seawater does not significantly affect its activity.

By varying the concentration of active ingredients within the formulation, a family of formulations result which are capable of responding to different hazardous situations. For rapid decontamination of surfaces, thin layers of foam may be sufficient, but strong active ingredient formulations are required. On the other hand, thick, uniform bubble size foam is an effective blast suppressant. High contamination is best handled with strong active ingredient formulations but the foam's structure, an important property for blast suppression,

1 can be somewhat compromised by high amounts of added ingredients, such as
2 decontaminants. At reduced amounts of active decontaminant, the foam's
3 structure remains unaltered, allowing it to be used for blast suppression, yet
4 retain decontamination abilities. Further, reducing active decontaminant and
5 buffer strength may also result in decreased corrosivity.

6 Accordingly, in a broad aspect of the present invention, there is
7 provided a family of decontaminant formulations comprising:

- 8 - from about 1% to about 15% by weight and preferably from
9 about 3% to about 9% by weight of a hydrated
10 chloroisocyanuric acid;
- 11 - from about 1% to about 10% and preferably from about 8%
12 to about 10% by volume of a co-solvent selected from the
13 group consisting of polypropylene glycols, polyethylene
14 glycols, and derivatives and mixtures thereof;
- 15 - from about 1% to about 15% and preferably from about 1%
16 to about 10% by volume of a surfactant;
- 17 - a buffer system to initially maintain said formulation at a pH
18 from about 8.5 to about 11 for a minimum of 30 minutes and
19 preferably initially, from about 10 to about 11; and
- 20 - the balance being water.

21 Preferably, the chloroisocyanuric acid is selected from the group
22 consisting of an alkali metal of monochloroisocyanuric acid and
23 dichloroisocyanuric acid such as sodium dichloroisocyanurate,
24 trichloroisocyanuric acid and a combination thereof with cyanuric acid. The

1 formulation may additionally comprise lithium hypochlorite to enhance the activity
2 of the dichloroisocyanuric acid salt.

3 In one preferred embodiment of the invention, the polypropylene
4 glycol has the chemical formula $R_1-(OCH(CH_3)CH_2)_n-OR_2$, where R_1 and R_2 are
5 independently H, an alkyl, or an ester group and $n>1$ or alternately, a partially
6 etherified polypropylene glycol where one of R_1 or R_2 is independently H, or an
7 alkyl group and $n>1$. In both cases the alkyl group may consist of a methyl, ethyl,
8 propyl, butyl or a mixture thereof. Use of certain higher molecular weight co-
9 solvents avoids subsequent false positive detection of the co-solvent as residual
10 contaminant by some monitoring equipment

11 Preferably, the buffer system forming the decontamination
12 formulation is a dual component inorganic buffer mixture of sodium tetraborate
13 decahydrate and anhydrous sodium carbonate adjusted to an initial pH of from
14 about 10 to about 11 using sodium hydroxide or, optionally, sodium metasilicate
15 pentahydrate.

16 One suitable surfactant consists of a composition of the formula
17 $[R(OCH_2CH_2)_nX]_aM_b$, where R is an alkyl group having from eight to eighteen
18 carbon atoms; n is an integer from 0 to 10; X is selected from the group of SO_3^{2-} ,
19 SO_4^{2-} , CO_3^{2-} and PO_4^{3-} , M is an alkali metal, alkaline earth metal, ammonium or
20 amine derivative; a is the valence of M and b is the valence of $[R(OCH_2CH_2)_nX]$
21 or a mixture thereof.

22 Preferably, the surfactant consists of a composition of the formula
23 $[R-CH=CH(CH_2)_m-X]_aM_b$ where R is an alkyl group having from eight to eighteen
24 carbon atoms; m is an integer from 0 to 3; X is selected from the group of SO_3^{2-} ,
25 SO_4^{2-} , CO_3^{2-} and PO_4^{3-} , M is an alkali metal, alkaline earth metal, ammonium or

1 amine derivative; a is the valence of M and b is the valence of $[R-CH=CH(CH_2)_m-$
2 X] or a mixture thereof.

3 Preferably, the surfactant also consists of a composition of the
4 formulae $R-OH$, where R is an alkyl group having from eight to sixteen carbon
5 atoms or mixtures thereof.

6 Preferably, the surfactant also consists of polypropylene glycol
7 having the chemical formula $R_1-(OCH(CH_3)CH_2)_n-OR_2$, where R_1 and R_2 are
8 independently H, an alkyl, or an ester group and $n>1$ or alternately, a partially
9 etherified polypropylene glycol where one of R_1 or R_2 is independently H, or an
10 alkyl group and $n>1$.

11 In another broad aspect of the present invention there is provided a
12 method of preparing and delivering a decontamination formulation comprising the
13 steps of:

- 14 - preparing a first aqueous solution comprising about 30% by
15 weight of chloroisocyanuric acid salt or the equivalent active
16 chlorine content of a mixture of chloroisocyanuric salt and
17 lithium hypochlorite;
- 18 - preparing a second aqueous solution comprising a mixture of
19 sodium tetraborate decahydrate, anhydrous sodium
20 carbonate, adjusted to a pH of from about 10 to about 11;
- 21 - providing a co-solvent selected from the group consisting of
22 polypropylene glycol, polyethylene glycol and a derivative
23 and mixture thereof;
- 24 - providing a surfactant comprising a composition of the
25 formulae $[R(OCH_2CH_2)_nX]_aM_b$, where R is an alkyl group

1 having from eight to eighteen carbon atoms; n is an integer
2 from 0 to 10; X is selected from the group of SO_3^{2-} , SO_4^{2-} ,
3 CO_3^{2-} and PO_4^{3-} ; M is an alkali metal, alkaline earth metal,
4 ammonium or amine derivative; a is the valence of M and b
5 is the valence of $[\text{R}(\text{OCH}_2\text{CH}_2)_n\text{X}]$ or a mixture thereof;
6 preferably, a composition of the formula $[\text{R}-\text{CH}=\text{CH}(\text{CH}_2)_m-$
7 $\text{X}]_a\text{M}_b$ where R is an alkyl group having from eight to
8 eighteen carbon atoms; m is an integer from 0 to 3; X is
9 selected from the group of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and PO_4^{3-} ; M
10 is an alkali metal, alkaline earth metal, ammonium or amine
11 derivatives; a is the valence of M and b is the valence of $[\text{R}-$
12 $\text{CH}=\text{CH}(\text{CH}_2)_m-\text{X}]$ or a mixture thereof;
13 preferably, the surfactant also consists of a composition of
14 the formulae $\text{R}-\text{OH}$, where R is an alkyl group having from
15 eight to sixteen carbon atoms or mixtures thereof;
16 preferably, the surfactant also consists of polypropylene
17 glycol having the chemical formula $\text{R}_1-(\text{OCH}(\text{CH}_3)\text{CH}_2)_n-\text{OR}_2$,
18 where R_1 and R_2 are independently H, an alkyl, or an ester
19 group and $n > 1$ or alternately, a partially etherified
20 polypropylene glycol where one of R_1 or R_2 is independently
21 H, or an alkyl group and $n > 1$;
22 - providing source water; and
23 - pumping the formulation through an aeration nozzle to create
24 a decontamination foam.

1 In a preferred aspect of the present invention, the co-solvent and
2 surfactant are mixed together and pumped with the source water through a
3 pumping device. The first and second aqueous solutions are introduced into the
4 stream between the pump and the aeration nozzle for delivery as a foam.
5 Addition of the more erosive and corrosive active decontaminant and buffer to the
6 stream after the pump is advantageous as it prolongs pump life.

7 Alternatively, all of the ingredients may be premixed with source
8 water and pumped simultaneously through the pumping device and the aeration
9 nozzle, or may be introduced to the source water stream individually.

10

BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1a and 1b are schematic representations of a pre-mix and a staged mixing embodiment of the method of application of the decontamination formulation;

Figures 2a – 2c are graphs showing detection for mustard agent, a reference mustard sample and a reference diethyl malonate sample respectively according to the decontamination of a vehicle set forth in Example 2.

Figure 3 is a complete mass spectrum of the library mustard spectrum m/z 109 peak in the middle trace of Fig. 1;

Figure 4 is a graph showing the absence of mustard agent in air samples taken near the vehicle of Example 2 after it has been treated with a decontamination formulation of the present invention;

Figure 5 is a table of results demonstrating decontamination of GA, GB, GD and HD agents according to Example 3;

Figure 6 is a table of results demonstrating decontamination of VX agents according to Example 4;

Figure 7 is a table of results demonstrating neutralization of a CW agent simulant DFP according to Example 5;

Figures 8 – 10 are graphs demonstrating the neutralization of DFP over time, according to Example 5, and specifically for formulations comprising the active ingredients SD alone, SD and KBr, and SD and LiOCl respectively;

Figures 11a – 14 are graphs demonstrating the decontamination of mustard from of a vehicle according to Example 7, specifically gas chromatograph (GC)-mass spectral (MS) analysis of samples before decontamination, an MS library spectrum trace, the library search values, GC/MS

1 analysis of samples 10 minutes after decontamination, and semi-quantitative and
2 chronological results from 8 chemical agent real-time monitoring stations
3 deployed around the vehicle;

4 Figure 15 illustrates the removal of radioactive dusts from a vehicle
5 according to Example 8.

6

7 **DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT**

8 In the present invention, a decontamination formulation and means
9 for use are provided which incorporates the known active ingredient,
10 hypochlorite, in a uniquely buffered solution designed to be incorporated into a
11 foam for maximal and stable coating, including vertical surfaces, for a prolonged
12 period including NATO prescribed periods of 30 minutes.

13

14 **Active Ingredient**

15 The formulation contains as an active ingredient, sodium
16 dichloroisocyanurate. Other chloroisocyanuric acids, their alkali metal salts or a
17 combination of acids including trichloroisocyanuric acid are also suitable for use
18 as the active ingredient. As an example, alkali metal salts of
19 monochloroisocyanuric or dichloroisocyanuric acid or a combination of any of the
20 above salts with cyanuric acid may be used.

21 The formulation of the present invention contains from about 1% to
22 about 15%, and preferably from about 3% to about 9%, by weight, of the
23 hydrated dichloroisocyanuric acid salt. The formulation may additionally comprise
24 lithium hypochlorite to enhance the activity of the dichloroisocyanuric acid salt.

25

1 Co-Solvent

2 The formulation further comprises a co-solvent consisting of from
3 about 1% to about 10% and preferably 8% to about 10% by volume, of propylene
4 glycol, polyethylene glycol, or derivatives or mixtures thereof. The glycol co-
5 solvent improves the solubilization of the CW agents, particularly the relatively
6 water-insoluble mustards, and thickeners, in otherwise aqueous solutions.
7 Typically, efficient solubilization is obtained in the range from about 8% upwards,
8 whereas lower amounts will provide some solubilization properties to the
9 formulation.

10 In one preferred embodiment of the invention, the polypropylene
11 glycol has the chemical formula $R_1-(OCH(CH_3)CH_2)_n-OR_2$, where R_1 and R_2 are
12 independently H, an alkyl, or an ester group and $n>1$. The alkyl group may
13 consist of a methyl, ethyl, propyl, butyl or a mixture thereof. In one example, both
14 R_1 and R_2 are hydrogens. Alternatively, the polypropylene glycol is a partially
15 etherified polypropylene glycol derivative having the same formula R_1-
16 $(OCH(CH_3)CH_2)_n-OR_2$, but where only one of R_1 or R_2 is independently H, or an
17 alkyl group and $n>1$. Again the alkyl group representing R_1 or R_2 may be a
18 methyl, ethyl, propyl, butyl group or a mixture thereof. Use of certain higher
19 molecular weight co-solvents avoids subsequent false positive detection of the
20 co-solvent as residual contaminant.

21

22 Surfactant

23 The formulation further comprises from about 1% to about 15% and
24 preferably from about 1.5% to about 10%, by volume, of a surfactant. The
25 surfactant is soluble in an aqueous medium and, when aerated, creates a foam.

1 The amount of surfactant used varies with the amount of co-solvent, active
2 ingredient and buffer present. In the presence of optimum levels of co-solvent,
3 the preferred amount of surfactant is from about 6% to about 10%, by volume. On
4 the other hand, when no co-solvent is added and relatively low amounts of active
5 ingredient are present, the preferred amount of surfactant can be as low as 1.5%
6 by volume. The surfactant wets the surfaces to be decontaminated and creates
7 foam on dispensing, suitable for covering and adhering to vertical surfaces. In
8 the case of radioactive dusts, the surfactant encapsulates the dusts for removal
9 from the subject surface.

10 Briefly, the surfactant consists of a composition of either the formula
11 $[R(OCH_2CH_2)_nX]_aM_b$, where R is an alkyl group having from eight to eighteen
12 carbon atoms, n is an integer from 1 to 10; X is selected from the group of SO_3^{2-} ,
13 SO_4^{2-} , CO_3^{2-} and PO_4^{3-} ; M is an alkali metal, alkali earth metal, ammonium or
14 amine derivative; a is the valence of M and b is the valence of $[R(OCH_2CH_2)_nX]$
15 or more preferably, the formula $[R-CH=CH(CH_2)_m-X]_aM_b$ where R is an alkyl
16 group having from eight to eighteen carbon atoms; m is an integer from 0 to 3; X
17 is selected from the group of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and PO_4^{3-} ; M is an alkali metal,
18 alkaline earth metal, ammonium or amine derivative, a is the valence of M and b
19 is the valence of $[R-CH=CH(CH_2)_m-X]$ or a mixture thereof and an alkyl alcohol,
20 R-OH, where R is an alkyl group having from eight to sixteen carbons. One such
21 suitable surfactant is Silv-Ex™ made by Ansul Fire Protection described in US
22 Patent 4,770,794 issued to Cundasawmy et al. September 13, 1988. More
23 specifically, the Silv-Ex surfactant consists of 20% by weight of
24 $C_{10}H_{21}(OCH_2CH_2)_{2-3}SO_4^-Na^+$, 20% by weight of $C_{14}H_{29}(OCH_2CH_2)_3SO_4^-NH_4^+$, 5%

1 by weight of $C_{12}H_{25}OH$, 20% by weight of diethylene glycol monobutyl ether,
2 0.5% of corrosion inhibitors and 34.5% by weight of water.

3 Alternatively, surfactants which do not contain diethylene glycol
4 monobutyl ether are preferable as residuals, as this low molecular weight
5 constituent can be detected by some conventional decontamination monitoring
6 equipment (such as Graseby Ionics™ Chemical Agent Monitor or CAM) and are
7 thus interpreted falsely as positive detection of residual contaminant.

8 A suitable surfactant consists of a composition of alkyl ether
9 sulphate salt, an alkyl alcohol, an alpha olefin sulfonate, a co-solvent and water.
10 More specifically the surfactant is a composition having the component formulas
11 of $[R_nH_{2n+1}(OCH_2CH_2)_mSO_4^{2-}M]$, where R is an alkyl group having from eight to
12 fourteen carbon atoms, m is an integer from 2 to 3, and M is Na^+ or NH_4^+ , in
13 mixture with R-OH where $R=C_{10}-C_{14}$, in mixture with
14 $CH_3(CH_2)_nCH=CHCH_2SO_3Na$, in mixture with $HO(CH_2(CH_3)CHO)_nH$ (PPG of MW
15 about 425) where $n=5-49$ and most preferably 7. The components are in water.
16 In addition, corrosion inhibitors can be added in very small quantities.

17 Accordingly, a preferred composition of a suitable non-residual
18 surfactant (or NR-surfactant) consists of 30% weight/volume of the sodium salt of
19 an ether sulphate of the formula $CH_3(CH_2)_{11}(OCH_2CH_2)_3OSO_3Na$; 15.5%
20 weight/volume of a sodium olefin sulphonate of the formula
21 $CH_3(CH_2)_nCH=CHCH_2SO_3Na$ where $n=10$ to 12 ; 50% weight/volume of
22 polypropylene glycol solvent of the formula $H(OCH(CH_3)CH_2)_nOH$ where $n = 5$ to
23 9 ; 2% weight/volume of an alcohol $CH_3(CH_2)_nOH$ where $n = 8$ to 16 ; about 0.3%
24 by weight of corrosion inhibitors such as sodium tolyltriazole, ammonium
25 dimolybdate and sodium pentahydrate silicate and the balance being water, with

1 additional water being added to dissolve other components. Further, this NR-
2 surfactant is capable of generating foam of uniform bubble size, is capable of
3 coating vertical surfaces, is compatible with water, gray water and seawater as
4 the main solvent, and is readily removed following decontamination by rinsing
5 with water.

6 To lower the thixotropic gelling point of the surfactant, useful in a
7 wider range of environments, it has been found that the alcohol component
8 preferably comprises more C₁₂ than C₁₄ (i.e. n=11). It has been found that diluting
9 the surfactant 1:1 with water for storage and transport further lowers the gelling
10 point.

11 Alternatively, a combination of surfactants can be used for the
12 preparation of the decontamination formulation. For example, Silv-Ex may be
13 combined with the NR-surfactant, or an alternative formulation or a combination
14 of them with other surfactant ingredients such as sodium laureth sulfate, having
15 the formula $\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{OSO}_3\text{Na}$, sodium C14-16 alpha olefin
16 sulfonate having the formula $\text{RCH}=\text{CHCH}_2\text{--SO}_3\text{Na}$, and ammonium alcohol
17 ethoxysulfate having the formula $\text{C}_{8-10}\text{H}_{17-21}(\text{OCH}_2\text{CH}_2)_{2-3}\text{OSO}_3^-\text{NH}_4^+$.

18

1 Buffer

2 The decontamination formulation of the present invention further
3 comprises a buffer that temporarily maintains an initial pH in the range of 10 to
4 11, sufficient to enable hydrolysis of G-agents and mustards and favor oxidation
5 of the V-agents so as to produce non-toxic products. An initial pH in the range of
6 10 to 11 is sufficient to provide adequate hypochlorite ions for decontamination.
7 Subsequently, it is desirable that the buffer fail, allowing the pH to decrease
8 eventually to a more neutral pH to enable more efficient destruction of the BW
9 agents.

10 As the buffer fails and the pH drops to a more neutral pH,
11 hypochlorous acid becomes more prevalent as hypochlorite ions react with
12 available hydrogen ions. Hypochlorous acid is the more active species with
13 respect to the destruction of BW agents as neutral species are able to enter the
14 cell more easily. Should BW agent survive the initial decontamination, the BW
15 agent and decontamination formulation may continue to co-reside over time,
16 perhaps after rinsing, and, as the pH falls, BW agent decontamination continues
17 at an even more effective pH. Further, from an environmental standpoint, a more
18 neutral final pH of the decontamination formulation is less hazardous.

19 It is important to maintain the initial high pH over a prescribed
20 duration (such as a NATO designated duration of 30 minutes), to provide
21 sufficient hypochlorite ions to effect decontamination – favoring oxidation of VX
22 agent which avoids the formation of toxic hydrolysis byproducts, favoring
23 hydrolysis of G-agents, and favoring oxidation of HD agents and avoiding HD
24 reformation. Accordingly, the buffer must be capable of buffering the release of

1 HCl due to hydrolysis of the chloroisocyanuric salts by water. Most preferably,
2 the pH is maintained above 8.5 during the duration available for decontamination.

3 It has been determined that the most suitable buffering system is an
4 inorganic buffering system, adjusted to an initial pH in the range of 10 to 11.
5 Sodium salts, such as a mixture of sodium tetraborate decahydrate and anhydrous
6 sodium carbonate, are preferable since quaternary ammonium compounds result
7 in depletion of hypochlorite through reaction with the hydrolysis product of
8 hypochlorite, chloride ion.

9 The preferred solvent for the decontamination formulation of the
10 present invention is water, including gray and seawaters.

11 The decontamination formulation may further optionally include
12 small amounts (preferably <0.03%) of corrosion inhibitors such as sodium
13 tolyltriazole, ammonium dimolybdate and sodium pentahydrate silicate to improve
14 compatibility with use on metals.

15

16 Augmented Active Ingredients

17 The decontamination formulation may further optionally include
18 lithium hypochlorite to augment the active hypochlorite content of the solution
19 over a short term, thus providing a higher level of active species in the initial
20 stages after the addition of water. Preferably, lithium hypochlorite is present in
21 amounts in the range of from about 5 to about 10% by weight of the active
22 ingredient dichloroisocyanuric acid salt and taking into account that commercially
23 available lithium hypochlorite is normally only available as 30% pure.
24 Alternatively, small amounts of Super Tropical Bleach (STB) or High Test
25 Hypochlorite (HTH), below their solubilisation limits so that no solid or slurry

1 results, could serve somewhat the same function as the addition of lithium
2 hypochlorite.

3 The decontamination formulation of the present invention may
4 further optionally include inorganic/organic bromide to increase the reactivity of
5 the chloroisocyanuric acid and generate low levels of hypobromite and bromine
6 chloride.

7

8 Optional Embodiments

9 Three embodiments are briefly described as follows and are more
10 specifically disclosed in the following examples.

11 In a first embodiment of the present invention the decontamination
12 formulation contains 9% sodium dichloroisocyanurate, a buffer mixture containing
13 0.0125M sodium tetraboratedecahydrate and 0.1M anhydrous sodium carbonate
14 adjusted to a pH from about 10 to 11, using NaOH (full strength buffer), 9%
15 surfactant and a total of 8% co-solvent, including co-solvent contained in the
16 surfactant mixture. This formulation provides for maximal decontamination -
17 capable of decontaminating the broad spectrum of CW and BW agents, in the
18 liquid phase, in under 7 minutes, and provides foam production capable of
19 coating vertical surfaces. The concentration of active ingredient of this first
20 embodiment tends to compromise the performance of the resulting foam as a
21 suppressant of dispersion or blast devices, likely due to the higher co-solvent and
22 salt content.

23 In a second embodiment of the present invention, the
24 decontamination formulation contains 6% dichloroisocyanuric acid salt, full
25 strength buffer, 9% surfactant and a total of 8% co-solvent. This formulation

1 provides for good decontamination and increased foam stability for
2 decontamination of any agents or for clean up after a blast.

3 In a third embodiment of the present invention, the decontamination
4 formulation contains 3% dichloroisocyanuric acid salt, a buffer in which the
5 concentrations of the components have been reduced by 1/3 that described for
6 full strength buffer (2/3 strength buffer), 3% surfactant and no extra added co-
7 solvent. This embodiment, while it provides excellent blast suppression, provides
8 slower reacting decontamination capability.

9

10 Method of Application

11 The decontamination formulation can be prepared either as a liquid
12 or as foam. The preferred form is to create foam due to its ability to effectively
13 coat surfaces, including vertical surfaces and to suppress vapor emissions.

14 Having reference to Fig. 1a, the decontamination formulation of the
15 present invention can be prepared by first combining in a single source solution in
16 a plastic drum, water bladder or plastic container, at approximately the final
17 percentages, the active ingredient, co-solvent, buffer, the surfactant and fresh or
18 seawater. The source solution is then pumped to the contamination site. For
19 foam application, the formulation is applied using high to medium pressure
20 pumping equipment equipped with appropriate aeration nozzles.

21 Referring to Fig. 1b, in an alternate and staged method the active
22 ingredient and buffer are made up separately from the co-solvent and
23 surfactant/foam. This staged approach provides improved storage life after
24 preparation. The active ingredient can be made up in a single solution
25 concentrate of the highest achievable percentage soluble in water, about 30% by

1 weight total in water. It follows that the higher the weight percent of soluble
2 active ingredient, the less concentrate is required to be aspirated into the main
3 stream to achieve maximum decontamination. This solution is stable for several
4 hours. The buffer mixture is prepared in a second solution at or near the solubility
5 limits of each of the buffer salts and the pH adjusted to provide an initial pH of 10
6 to 11. This concentrate is stable for long periods of time. The active ingredient
7 and buffer can then be introduced, into a stream of co-solvent, surfactant and
8 water for completing the formulation and initiating decontamination.

9 The concentrations of co-solvent and surfactant are dependent on
10 one another and on the type of decontaminant applicator or inductor used. A
11 synergistic effect can exist between these two ingredients. As well, the ambient
12 temperature can influence the concentration of surfactant required. Therefore,
13 one must consider these factors and adjust the concentration of the surfactant to
14 suit the particular situation in which the formulation is to be used.

15 Regardless of the method of formation, most preferably, the
16 decontamination formulation is prepared by adding into a stream of water, the
17 ingredients in the following order; co-solvent and surfactant, active ingredient,
18 and buffer.

19 The ingredients are pumped through an appropriate aeration nozzle
20 to provide a relatively stable and thick foam. The nozzle should entrain sufficient
21 air into the stream to create the foam without causing excessive back pressure.

22 The active ingredient and the buffer are added as concentrates to
23 the stream of water and are diluted during the application process. The surfactant
24 can be added simultaneously with the buffer, however it may be advantageous to

1 add them separately (Fig 1b) as the amount of surfactant required depends upon
2 the ambient temperature, the surface being treated and the incident sunlight.

3 By adding the surfactant separately, wholly or as an optimizing
4 addition to a solution already containing most of the decontamination ingredients,
5 one can beneficially adjust the foam properties to the ambient conditions.

6 One further advantage to the staged approach is that hypochlorite
7 or buffer are introduced to the stream after the pump and before the nozzle so
8 that the pump is only exposed to water or possible pump-friendly co-solvent and
9 surfactant. Greater pump life can be expected as it is not degraded or corroded
10 by long-term exposure to potentially corrosive or abrasive ingredients.

11 In the alternative approach, all ingredients are combined in the
12 source container (Fig. 1a). While this approach is simpler, it must be noted that
13 the presence of the buffer mixture will immediately initiate degradation of the
14 active ingredient so the lifetime of the formulation using this method of
15 preparation may be more limited from the time at which they are mixed.
16 Additionally, the pump will be exposed to the complete formulation and could
17 corrode substantially faster, depending upon the materials of construction. In
18 contrast, the lifetime of the active ingredient in water without the addition of the
19 buffer mixture (Fig. 1b) is considerably longer.

20 Modifications to the above methods are possible. For example, the
21 solutions could be mixed off-line in a series of drums or tanks and, when
22 dissolved, the contents could be pumped to source containers permanently
23 attached to the pumps or aspirators.

24

1 Kit

2 For field use, a practical approach is to provide appropriate
3 quantities of each component in kit form and obtain a local source of water.
4 Separate, lightweight containers such as plastic pouches or pails facilitate
5 transport of the components to the decontamination site. For example, the active
6 ingredient, which is in the form of a powder, can be weighed out in specific
7 amounts and heat-sealed in a plastic pouch to keep it dry. Similarly, the buffer
8 components, also available as solids, could be packaged individually or as a
9 mixture with the active ingredient if moisture can be excluded.

10 The co-solvent can likewise be measured out in appropriate
11 quantity, diluted slightly if necessary and stored in large plastic pails with tightly
12 sealed lids. The surfactant can likewise be supplied in its original shipping pail or,
13 if prepared locally, stored in pails in pre-measured amounts similar to the co-
14 solvent. Alternatively, the co-solvent and surfactant can be provided as a mixture
15 and packaged together. The solid ingredients are then dissolved into solution in
16 water or seawater, which are subsequently added to a pumping system as
17 described above to obtain the decontamination formulation of the present
18 invention at the decontamination site.

19

1 Examples

2 The following examples are illustrative of the preferred
3 embodiments of the present invention and are not to limit the scope of the
4 invention.

5 Example 1 illustrates typical preparation of a decontamination
6 formulation.

7 Example 2 illustrates the application and effectiveness of the
8 formulation of Example 1 as applied in a field trial for destruction of a mustard
9 chemical agent.

10 More generally, examples 3 through 5 illustrate various formulations
11 and results for liquid phase reaction-decontamination of CB agents. Specifically,
12 examples 3 and 4 illustrate liquid phase reaction-decontamination of G-Type
13 Nerve and Mustard Agents and VX Nerve Agent.

14 Example 5 similarly illustrates liquid phase reaction-
15 decontamination of a known nerve agent simulant, di-isopropyl fluorophosphate
16 (DFP).

17 Example 6 illustrates the foam phase-detoxification of viable
18 anthrax spores on military-spec painted metal coupons.

19 Examples 7 and 8 demonstrate field trial results for the
20 decontamination of a military vehicle, particularly the destruction of mustard
21 chemical agent and foam phase removal of radioactive dusts.

22

1 Example 1

2 The following decontamination formulation was prepared for the
3 vehicle decontamination according to Example 2.

4 A source solution of water, buffer, co-solvent and surfactant was
5 prepared. Separately, a solution of active ingredient was prepared. Separate
6 preparation of the active ingredient postpones the initiation of the degradation of
7 the hypochlorite precursor until mixed.

8 More particularly, a concentrate of the active ingredient was
9 prepared from 72 liters of tap water and 18.6 kg of anhydrous sodium
10 dichloroisocyanurate. The solid active ingredient was added to the water in a
11 plastic waste overpack container and vigorously stirred with an industrial
12 stirrer/homogenizer. The solution turned into an off-white milky liquid which,
13 when gently warmed with the introduction of steam for less than five minutes
14 turned into a translucent amber-colored fluid. Mechanical constraints for this
15 particular experiment limited the solution concentration to a maximum of 5.6%
16 active ingredient, 9% being achievable using different equipment as
17 demonstrated in Examples 3 - 5.

18 The source solution was prepared with 303 liters of tap water, 16.73
19 liters of surfactant, 26.35 liters of PPG 425 as co-solvent and inorganic buffer
20 salts, more particularly, sodium tetraborate decahydrate and anhydrous sodium
21 carbonate in sufficient amounts to provide concentrations of 0.0125M and
22 0.1000M respectively in the final solution. Sodium hydroxide was added in
23 sufficient amounts to provide an initial pH of approximately 11, which would, after
24 addition of the active ingredient, cause the resulting pH after stabilization to be
25 from about 9.3 to about 9.7.

1 An NR-surfactant, modified from the Silv-Ex formulation, was used.
2 Generally the composition of the NR-surfactant was, all referenced by weight,
3 30% $C_{8-10}H_{17-21}(OCH_2CH_2)_{2.3}OSO_3^-NH_4^+$, 15.5% $C_{11-13}H_{23-27}CH=CHCH_2-SO_3^-Na^+$,
4 20% PPG 425, 5% alcohol mixture (of about 2% $CH_3(CH_2)_{11}OH$ and 3%
5 $CH_3(CH_2)_{13}OH$, and the balance being water.

6 Note that the NR-surfactant already contained 20% by weight of co-
7 solvent and thus only sufficient additional co-solvent (26.35 liters) was added to
8 the source solution to obtain an 8% overall solution (29.75 liters).

9 The source solution and concentrate were separately stored in two
10 plastic storage vessels. The source solution was pumped at 24 liters/min through
11 pressure hose to a foam nozzle. The concentrate was introduced into the flow of
12 source solution immediately downstream of the pump, through two eductors
13 backed by small centrifugal pumps whose flow rates were constantly monitored.
14 The combined eduction of the two units amounted to a total of 18.6% of the
15 overall exit flow of foamed effluent from the nozzle. This combination provided a
16 final active ingredient concentration of approximately 5.6% by weight equivalent
17 of sodium dichloroisocyanurate dihydrate. Two eductors were provided in
18 anticipation of alternate operation wherein each eductor would draw in a separate
19 concentrate; one containing active ingredient, the other containing the buffer, co-
20 solvent, and possibly, the surfactant components.

21 In operation, the combined effluent was fed through 40 m of
22 standard high-pressure hose to a spray lance. Dissemination was achieved
23 through attachment of a foam nozzle (9 US Gal/min) to the spray lance
24 discharge. As a result, foam was readily generated by pumping the formulation

1 through the system and applying the spray from the nozzle to the sides of the
2 target vehicle.

3

4 Example 2

5 Using the formulation as set forth in Example 1, neutralization of
6 mustard agent applied to a vehicle surface was evaluated in the field as follows.
7 Approximately 150 ml of mustard was applied to the surface of a vehicle using a
8 paintbrush. The presence of mustard agent was assessed and verified using a
9 portable gas chromatograph/mass spectrometer (GC/MS). The decontamination
10 formulation of Example 1 was applied to the contaminated side of the vehicle
11 using the lance and nozzle followed by manual scrubbing of the surface using
12 long-handled brushes. After a 30 minute wait period, the foam was washed away
13 with water and the vehicle surface was re-surveyed using the GC/MS. Fig. 2
14 illustrates that an air sample taken near the contaminated vehicle before
15 decontamination contained mustard agent, the top trace is the total ion current as
16 recorded by a portable GC/MS which shows two large peaks due to internal
17 standards (IS) and two lower peaks. The second trace (Fig. 2b) is an ion
18 chromatogram set at m/z 109 and the bottom trace (Fig. 2c) is a separate ion
19 chromatogram set at m/z 115 to detect a simulant, diethyl malonate, also present
20 in the atmosphere from an earlier contamination. As shown in Fig. 3, a mass
21 spectral analysis of the m/z 109 sample component of Fig. 2b confirmed that this
22 component was mustard chemical agent with a 85.7% probability as compared to
23 the bottom trace, which is an authentic mass spectrum of mustard stored in the
24 search library. Turning to Fig. 4, once the vehicle was treated with the

1 decontamination formulation, no further mustard was detected in air samples
2 taken near the vehicle.

3

4 Examples 3 – 5

5 In each of Examples 3 - 5, quantitative analyses for residual agents
6 were performed on a high pressure liquid chromatography (HPLC) system for
7 separation of the reaction components, equipped either with a HPLC-UV detector
8 in series with a commercially available dual flame gas chromatographic flame
9 photometric detector (FPD) from Varian Associates, or, where possible, on a
10 Hewlett-Packard 1100 LC-MS system equipped with a diode-array UV-VIS
11 spectrophotometer and mass selective detector (MSD). The water used in the
12 reactions, prepared solutions, and in the HPLC was distilled and deionized. The
13 formulation for the surfactant/foam was first warmed to 32°C to ensure
14 homogeneity. CB agents and simulant DFP were provided by the Canadian
15 Single Small Scale Facility at the Canadian Defence Research Establishment
16 Suffield (DRES) in southern Alberta, Canada and Aldrich Chemical Company,
17 respectively. GB stock calibration solution was prepared by weight in acetonitrile
18 (AcCN) and several dilutions were prepared ranging from 25 to 900 ng/μL for
19 calibration of the FPD, UV, and MSD responses. Stock solutions of the other CW
20 agents were prepared volumetrically in AcCN and similarly diluted for calibration.

21 Unless otherwise specified, in a typical experiment, samples were
22 prepared in 2.0mL autosampler vials. The first addition was a water solution
23 containing the surfactant and, if necessary, the co-solvent. This was followed by
24 buffer concentrate, then the decontaminant concentrate which had been
25 separately prepared by adding the active ingredient, anhydrous sodium

1 dichloroisocyanuric acid (SD), to water and heating to 29°C with stirring for 15-30
2 minutes. Finally, the CB agent was added defining time zero, and aliquots, at
3 noted elapsed times, were directly injected into the LC. The temperature of the
4 vial holder was maintained at 25.0°C and a mini stirbar in the vial mixed the
5 components. Fresh samples were prepared for each FPD analysis to obtain
6 residual agent concentration profiles over time and these same solutions were
7 subsequently analyzed by LC-MS.

8

9 Example 3

10 Having reference also to Fig. 5, the effectiveness of several
11 decontaminant formulations against selected G-type nerve gases GB, GA and
12 GD and mustard gas, HD, was determined. The formulations tested consisted of
13 an active ingredient, a surfactant, an inorganic buffer mixture and, optionally, co-
14 solvent, in excess of that already present in the surfactant mixture. The co-
15 solvent values in Fig. 5 represent added co-solvent and that contained in the
16 surfactant.

17 Three decontamination formulations were assessed for
18 effectiveness against typical G-nerve agents; the mildest formulation, using 3%
19 w/w SD, a 2/3 strength buffer, and 1.3% w/w surfactant; an intermediate strength
20 formulation with 6% w/w SD, full strength buffer, 4.6% w/w surfactant and an
21 additional 6.9% w/w to 7.8% w/w co-solvent, and a full strength formulation with
22 9% w/w SD, full strength buffer, 4.8% w/w surfactant and 6.9% w/w additional co-
23 solvent. Although anhydrous SD was used in preparation of the solution,
24 percentages are quoted in terms of the equivalent amount of dihydrate.
25 Percentages (w/w) quoted for surfactant represent double-strength surfactant.

1 In order to standardize concentrations between experiments, the
2 effectiveness was calculated as a percentage of residual agent.

3 Using 0.29% w/w GB, there was no evidence of residual agent in
4 any of the LC-FPD or LC-MS analyses for the mildest and intermediate strength
5 formulations (3% w/w and 6% w/w SD). GB was destroyed in each case before
6 the first sample could be taken (0.43 and 1.13 minutes respectively). For the
7 most potent formulation (9% w/w SD), only LC-FPD analysis was performed at
8 1.78 minutes elapsed time and no agent was detected indicating complete
9 destruction of the agent within 1.78 minutes.

10 Using 0.29% w/w GA, only the mildest and intermediate strength
11 formulations (3% w/w and 6% w/w SD) were evaluated. The mildest formulation
12 was tested in two separate experiments. In the first, containing ~1.6% w/w
13 surfactant, LC-FPD analysis indicated that GA was destroyed within 1.33
14 minutes. In the second, containing ~1.8% w/w surfactant, there was no evidence
15 of GA in 1.07 minutes elapsed time (LC-FPD) or 3.43 minutes (LC-MS). For the
16 intermediate strength formulation containing an additional 7.5% w/w co-solvent,
17 there was no evidence of GA in 1.07 minutes elapsed time by LC-FPD or 3.35
18 minutes by LC-MS.

19 Using 0.29% GD, again only the mildest and intermediate strength
20 formulations were each evaluated. The full strength formulation was not tested
21 due to the success with the two milder formulations. The mildest formulation was
22 tested and, in contrast to the other two G-agents examined, small amounts of
23 residual GD appeared to be observed for the shortest reaction time sample.
24 Specifically, as analyzed by LC-FPD, 5.0% residual agent appeared to be
25 present at 1.07 minutes and 0.5% appeared to remain at 4.77 minutes, and the

1 agent was completely gone by 10 minutes, as determined by LC-MS analysis.
2 Similar results were observed using the intermediate solution containing 7.8% co-
3 solvent. Complete LC-MS characterization of the peak eluting at GD in a stock
4 solution of GD suggests that a trace of a GD-related impurity,
5 methylpinacolylmethylphosphonate also eluted at this point, possibly contributing
6 to the residual peak observed at short reaction times. Thus, although GD appears
7 to be more difficult to destroy than GB or GA, the mildest formulation is still very
8 effective against GD within acceptable time limits.

9 Using 0.27% w/w HD, again due to their success, only the mildest
10 and intermediate strength formulations were evaluated. The mildest formulation
11 was tested for effectiveness against HD in three separate tests. In the first test,
12 there was no evidence of residual HD after 2.67 or 4.92 minutes (reaction
13 solutions had to be mixed more vigorously than the other agents due to limited
14 solubility of HD so earlier sampling was not possible). In the second test, no
15 residual agent was detected after 3.0 or 62.1 minutes, however 6.2% of residual
16 HD appeared to be present after 5.4 minutes assuming that the eluting peak was
17 indeed HD. As a confirmatory test, an third experiment was performed and no
18 HD was detected after 3.65 or 4.97 minutes.

19 It is therefore concluded that the mildest formulation is completely
20 effective against this level of HD in less than 2.7 minutes.

21 The intermediate formulation also tested for effectiveness against
22 HD and demonstrated no residual HD after 2.47, 5.27, or 53.3 minutes.
23 Verification by LC-MS could not be performed as HD cannot be detected using
24 positive API-ES under these conditions.

25

1 Example 4

2 Having reference also to Fig. 6, the effectiveness of several
3 formulations against the nerve agent VX was determined.

4 Samples were prepared as described in Example 3. Two
5 decontaminant formulations were assessed for effectiveness against VX-nerve
6 agent: the mildest formulation (MILD) with 3% w/w SD, 2/3 strength buffer, and
7 1.3% w/w surfactant, and the full strength formulation (FS*) with 9% w/w SD, full
8 strength buffer, 4.8% w/w surfactant and 6.9% w/w additional co-solvent. As with
9 example 3, percentages quoted for surfactant represent double-strength
10 surfactant.

11 Control formulations were also examined. These included a
12 formulation containing only full strength buffer and surfactant (Buffer/Surf) and a
13 formulation containing all ingredients of the full strength decontaminant but
14 without active ingredient (FS*wo/SD).

15 In order to standardize concentrations between experiments,
16 effectiveness was calculated as percentage of residual agent. In addition, an
17 authentic sample of a known potential toxic product (Toxic Product), of hydrolysis
18 of VX, S-(2-diisopropylaminoethyl) methylphosphonothioic acid was synthesized
19 and characterized by LC-MS to be used as an indicator of unsuccessful
20 detoxification of VX. All reaction mixtures were examined for the presence of
21 this compound; the presence of significant quantities would be sufficient evidence
22 to disallow the formulation as a possible decontaminant candidate. The results
23 are summarized in Fig. 6.

24 In the first evaluation, the control formulation of buffer and
25 surfactant (Buffer/Surf) was tested at a low concentration of VX (4 μ L/mL). After

1 six days, 42% of the VX remained and toxic product in significant quantity was
2 detected. The control formulation of full strength formulation without active
3 ingredient (FS*wo/SD) was tested against a concentration of 12 μ L/mL of VX.
4 Again, significant quantities of VX and toxic product were found at 125 minutes
5 and 6 days. Additionally, there was evidence of VX droplets in the solution at 125
6 minutes indicating that saturation levels of VX were present in solution and that
7 removal of VX from the system was slow. When full strength formulation with SD
8 was employed in excess (18.2:1 active species/VX), all VX was destroyed in less
9 than 7 minutes with no evidence of toxic product.

10 A more extensive examination of the temporal effectiveness of the
11 mildest formulation was undertaken in which the stoichiometric ratios of
12 concentrations of VX to active chlorine present in solution were varied. For the
13 lowest ratio (~6:1), effective decontamination of VX was not achieved although
14 only small traces of toxic product were observed. On the other hand, if the ratio
15 was ~16-18:1, complete decontamination without significant production of toxic
16 product was achieved. As shown in Fig. 6, the mildest formulation at a ratio of
17 18.2:1 is completely effective in less than eleven minutes. A similar formulation
18 reacting at a ratio of 29:1 resulted in similar effectiveness, however this is most
19 likely due to the fact that the trace recorded by the LC-MS is at its detection limit
20 using this procedure.

21 An analysis of the mild formulation without added VX did not
22 register any response for VX eliminating the possibility of a false positive VX
23 result due to the formulation itself.

24 In conclusion, even the mildest formulation is highly effective
25 against VX provided that the ratio of reactant to agent is maintained over at least

1 17:1. This finding is in accordance with statements made in Y-C Yang, J.A.
2 Baker, and J.R. Ward, Chem Rev., 1992, 92, p1731, in which the authors state
3 that greater than 10 moles of active chlorine are required to oxidize 1 mole of VX.
4

5 Example 5

6 Having reference also to Fig. 7, the effectiveness of several
7 decontaminant formulations was tested against diisopropylfluorophosphate
8 (DFP), a compound often employed as a simulant for G-type nerve gases.
9 Formulations in which the active ingredient, sodium dichloroisocyanurate (SD),
10 was augmented by lithium hypochlorite (30% LiOCl) and potassium bromide
11 (KBr) were also tested. As with the previous examples, the percentages quoted
12 for surfactant represent double- strength surfactant.

13 Following introduction of surfactant and, if applicable, co-solvent,
14 active ingredient (SD) was added as a 30% concentrate prepared in distilled,
15 deionized water by adding solid SD to a measured amount of water which was
16 then heated to 29°C, with stirring, for 20-30 minutes. When SD/LiOCl
17 combinations were used, a concentrate was prepared and added to the reaction
18 solution in a similar manner. Constant pH was maintained at 9.5 using an
19 automatic titrator adding dilute NaOH. As a final step, the DFP was weighed out
20 and added to the solution, defining time zero for the reaction. At timed intervals
21 (5, 10, 15, 30, 60 and 120 minutes), aliquots were taken from the reaction
22 solution, delivered into a quench vial containing aqueous hydrogen peroxide in
23 methanol or isopropanol, and the aliquot weight recorded, along with the exact
24 time of the quenching. Each quenched sample was then analyzed by HPLC.

1 In order to standardize concentrations between experiments, the
2 effectiveness was calculated as percentages of residual DFP.

3 The results and experimental parameters are summarized in the
4 table of Fig. 7 and the graphs of Figs. 8 - 10. The table of Fig. 7 is divided top
5 down into three sections representing the three formulations of SD, SD+KBr, or
6 SD+LiOCl respectively.

7 In the first formulation (SD) and having reference to Figs. 7 and 8,
8 the results of a control containing no active ingredient, surfactant or co-solvent
9 and various formulations of SD, co-solvent and surfactant, at pH 9.5, are
10 illustrated.

11 As a control for comparison purposes, and entitled test 7-115, the
12 disappearance of DFP in aqueous solution at pH 9.5 by unaided hydrolysis was
13 monitored. The % of DFP remaining over time is plotted on Fig. 8 as line 81
14 wherein the control indicated an apparent initial increase in the DFP followed by a
15 decrease over time to a value of 68% at 107 minutes. The calculated percentage
16 of DFP remaining at 30 minutes was 87%.

17 The DFP response for a similar test 7-97 at pH 9.5, in which only
18 SD was added, is plotted on Fig. 8 as line 82 and illustrates a rapid drop over
19 time to a value of zero at approximately 30 minutes.

20 In two additional tests, 7-123 and 7-137, co-solvent PPG425 (7.2 %
21 w/w and 7.9% w/w respectively) was added to the water, held at pH 9.5 in the
22 reaction vessel and stirred. SD (7.97% w/w and 7.42% w/w respectively) and
23 finally DFP (1.34% w/w and 1.25% w/w respectively) were added. Test 7-137
24 was performed with the addition of surfactant (3.5% w/w) along with the co-
25 solvent.

1 Plotted as lines 83 and 84 respectively, there was an exponential
2 decrease of the percentage of residual DFP with time. However, the curve is
3 shifted upwards from that of the reaction of SD alone with DFP at pH 9.5, and, in
4 fact, the DFP was not destroyed in two hours. At the 30-minute mark, 25% of the
5 DFP remained in the reaction solution with co-solvent alone and 21% with the
6 addition of surfactant and co-solvent.

7 It is clear that the addition of SD, whether alone or in the presence
8 of co-solvent and surfactant significantly increases the rate of hydrolyses of DFP
9 but that both of these other additives has a negative effect on reaction rate.

10 In the second formulation and having reference to Figs. 7 and 9, the
11 results for controls and the effect of augmenting the active ingredient with the
12 addition of KBr to SD with co-solvent and surfactant is demonstrated. Since the
13 addition of co-solvent and NR-surfactant demonstrated a retarding effect on the
14 rate of hydrolysis of DFP, the ability of KBr when added to the SD to offset this
15 effect was investigated. As a control, the results from a test 7-143, plotted as line
16 91, with both co-solvent (6.3% (w/w)) and surfactant foaming agent (3.4% (w/w))
17 in the reaction solution were compared to a similar reaction at pH 9.5 involving
18 added KBr. In the control case, there was residual DFP after two hours and 23%
19 remained after 30 minutes.

20 In test 7-147, a KBr (0.1 M) solution held at pH 9.5 was substituted
21 in place of water and the disappearance of DFP was determined. As plotted line
22 92 shows, although the initial value at five minutes appears to be anomalously
23 low (12% DFP) since the DFP appears to increase to 26 % at 10 minutes then
24 gradually decrease with time, it is clear that the rate of hydrolysis of DFP has
25 increased relative to the control formulation. The DFP did not reach zero within

1 one hour and the calculated percentage of DFP remaining at 30 minutes was 8%.
2 Clearly, addition of KBr assists in the rate of hydrolysis of DFP in the presence of
3 surfactant and co-solvent.

4 In the third formulation and having reference to Figs. 7 and 10, the
5 results of a control and a formulation augmented by the addition of LiOCl to the
6 SD are illustrated. As an alternative to adding relatively insoluble KBr for
7 increasing overall hydrolytic reactivity, a soluble hypochlorite, LiOCl, was
8 substituted. As a comparison, a first test, plotted as line 101, was performed in
9 which the reactivity of SD (11.32%(w/w)) in a solution containing surfactant (1.5%
10 (w/w)) and being held at pH 9.5 was examined. The DFP decreased with time
11 until it was undetectable after 0.5 hours. The calculated percentage of DFP at 30
12 minutes was 2%.

13 In a second test 8-23, plotted as line 102, LiOCl at 0.19% w/w was
14 added to a solution with SD (6.52 %) and the surfactant (1.5% (w/w)) and
15 maintained at pH 9.5. The weight of the LiOCl was 3.0% of the SD. The DFP at
16 five minutes was less than 20% of the initial value and continues to decrease with
17 time. The calculated percentage of DFP remaining at 30 minutes is only 0.5%.

18 Clearly, the substitution of LiOCl to a lower concentration of SD
19 leads to a solution with more reactivity toward DFP than one with SD as the only
20 active ingredient. Also, when compared to Example 3 above, it is apparent that
21 DFP is much more resistant to hydrolysis in this system than are the G-agents it
22 was proposed to simulate.

23

1 Example 6

2 The effectiveness of foam phase-detoxification of anthrax spores
3 was determined. A suspension of *Bacillus anthracis* (Ames strain) was heat
4 shocked to kill the vegetative cells, leaving only the viable spores. Small metal
5 coupons, painted as per in-service military vehicles, were cleaned with ethanol
6 wipes and sterilised by autoclaving. Each coupon to be used was spotted with
7 200 µL spore suspension, distributed over the surface of the coupon as 60-70
8 small droplets and allowed to dry overnight in a biosafety cabinet in a Level 3
9 Biocontainment laboratory.

10 Two trials were performed on two separate days using freshly
11 prepared foam formulations. Each trial used two of these coupons, one to test the
12 decontamination formulation and one to act as a control. Each coupon was
13 placed in a 100 mm petri dish, supported to keep it from coming in contact with
14 the bottom of the dish and covered with either the decontamination foam of the
15 present invention or a control foam not containing the decontaminant active
16 ingredients. The lid of the petri dish was replaced and twisted to ensure that the
17 foam contacted the entire coupon. After 30 minutes each coupon was removed
18 from the petri dish using forceps, rinsed with sterile PBS, then swabbed twice
19 over its entire surface with a sterile sampling swab. The swab was placed in 5 ml
20 of Heart Infusion broth and vortexed.

21 In both trials, 200 µL of neat broth from the decontamination foam-
22 treated coupon and 200 µl of a 1×10^{-4} dilution (in PBS) of the broth from the
23 control foam-treated coupon were plated onto each of four Blood Agar plates.
24 The plates were incubated overnight at 37° C and the Colony Forming Units

1 (CFU) observed the following day, are given in Table II. The Control foam results
2 are shown multiplied by 10^4 to adjust for the 10^{-4} dilution.

3 Trial 1 and Trial 2 indicate, respectively, that, on average, only
4 0.0108% and 0.00109% of the original material on the decontamination foam-
5 treated coupons remained viable, translating into a 99.989% and 99.999% kill for
6 simple contact with the decontamination foam for a period of 30 minutes.

7

8 Table II - Data from Anthrax Spore Decontamination Trials.

Experiment	Colony Counts			
	Plate 1	Plate 2	Plate 3	Plate 4
Trial 1 – Decon foam	33	26	28	21
Trial 1 – Control foam	22×10^4	22×10^4	29×10^4	28×10^4
Trial 2 – Decon foam	13	10	5	3
Trial 2 – Control foam	66×10^4	72×10^4	68×10^4	78×10^4

9

10 Example 7

11 Having reference to Figs. 11 – 13, the neutralization of mustard
12 chemical agent on a military vehicle surface was evaluated in a field trial using a
13 formulation comprising of a mixture of sodium dichloroisocyanurate and LiOCl as
14 active ingredients. The vehicle used was a US M113A armored personnel carrier
15 subsequently coated with Canadian Forces specification Chemical Agent
16 Resistant Coating (an agent-resistant two-pot polyurethane paint).

17 In this decontamination trial approximately 75 mL of munitions-
18 grade mustard agent was painted onto the side and end of the vehicle. The
19 vehicle was located inside a plastic-lined containment pit.

20 In Fig. 11, a mass spectral analysis of the total ion and
21 reconstructed m/z 109 chromatograms confirmed that the contaminant in the
22 bottle and painted onto the vehicle was, indeed, mustard by reference to an

1 authentic mass spectrum of mustard stored in the search library (Figs. 11a, 12).
2 Handheld Chemical Agent Monitors (CAMs) exhibited strong H-mode mustard
3 responses and 3-Way Detector Paper displayed the characteristic red colour
4 response indicative of blister agents when pressed onto the contaminated
5 surface of the vehicle.

6 Referring to Fig. 13a, the decontamination formulation was then
7 applied to the contaminated vehicle using a high capacity pump and two hoses
8 fixed with foam nozzles. The vehicle was then scrubbed using long-handled
9 brushes. During and following these steps, readings were made of the air around
10 and downwind of the vehicle. Immediately, Chemical Agent Monitor (CAM) and
11 air sample surveys conducted around the vehicle during the scrubbing procedure
12 failed to detect the presence of mustard vapour, as shown by the GCMS results
13 of Fig. 13a (total in chromatogram) and Fig. 13b (m/z 109 reconstructed mass
14 chromatogram characteristic of mustard) compare to the corresponding traces in
15 Fig. 11.

16 Following a short (<30 min) rest period, the foam was washed away
17 with water and the air near the vehicle surface surveyed against using CAMs and
18 the GCMS. Once the vehicle had been treated, CAM surveys conducted close to
19 the vehicle surface showed no response, indicating mustard vapour was not
20 present.

21 Having reference to Figure 14, the combined responses from four
22 Chemical Agent Detection Systems Mark II (CADS II) stations deployed around
23 the vehicle are illustrated. Each CADS II station comprises two CAMs. In this
24 figure, the readings of all eight CAMs (four CADS II stations X 2) were summed
25 and displayed. The vertical bars in the figure denote significant actions on the

1 part of trial personnel. Gross contamination of the vehicle was initiated at point A
2 and decontamination commenced at point B. By point C, the audible alarm from
3 the CADS II central control unit (CCU) had silenced and from point D onward, no
4 further detection or bar reading of mustard vapor was observed. Thus, this
5 formulation applied in this manner is effective in suppressing agent vapor from a
6 freshly contaminated-coated military surface immediately and is effective in
7 decontaminating mustard-contaminated military vehicles within a 30-minute
8 period after application.

9

10 Example 8

11 Having reference to Fig. 15, the effectiveness of the foaming agent
12 by itself to effect decontamination of radioactive dusts from the exterior surface of
13 an armored vehicle was demonstrated. The vehicle, a French AMX-10 Armored
14 Personnel Carrier, was contaminated by spraying the exterior with ^{140}La particles
15 (100-200 μm) to simulate surface contamination as might be caused by driving
16 across contaminated dusty terrain. Decontamination formulation using Silv-Ex
17 surfactant was sprayed over the surface of the vehicle using a powered pressure
18 washer fixed with an air induction foam nozzle of the type normally used in
19 applying fire-fighting foams. Subsequent to the application of decontaminant, the
20 vehicle was towed to a sensing frame where radiation measurements on the
21 exterior could be made. In Figure 15, the radiation level measured inside the
22 vehicle in the first trial was observed to be in the order of 30 mRem/hr. After
23 towing to the decontamination site and commencing application, the radiation
24 level was observed to drop significantly (to approximately 11 mRem/hr)
25 presumably due to foam layers dropping off the sides of the vehicle during the

1 application stage. The radiation level flattened off over the course of the
2 decontamination probably due to residual particles remaining on the vehicle in
3 areas where the foam could not drop off (top, crevices) readily. On
4 commencement of rinsing of the vehicle with water, the radiation level dropped
5 even further (to approx. 6 mRem/hr) presumably due to flushing off some of the
6 remaining radioactive particles. A map of the radiation emitted from the exterior
7 surface of the vehicle as sampled by a frame of 80 probes confirmed that the
8 radiation had been significantly reduced by decontamination using Silv-Ex-based
9 decontamination foam.

10 In a subsequent trial, the same vehicle was contaminated to a level
11 of approximately 45 mRem/hr. During movement of the contaminated vehicle to
12 the site of decontamination, significant loss in the level of radioactivity was
13 observed. The loss was such that the trial was terminated. It was apparent that
14 the exterior surface, having been previously cleaned in an earlier trial, did not
15 retain radioactive particles sprayed onto it. In other words the surface had been
16 degreased and dust adherence had been significantly decreased, suggesting an
17 additional benefit to the use of the formulation.

18 In a related examination in which paint panels were contaminated
19 and subsequently decontaminated by dry scrubbing, the standard approach for
20 decontamination of radioactive particulate matter was observed to attain a low
21 level of 0.55 mRem/hr whereas decontamination with Silv-Ex-based
22 decontamination foam reduced the radiation to a level of 0.33 mRem/hr after one
23 application and 0.22 mRem/hr after a second decontaminant application, both of
24 which surpass the standard approach for addressing this hazard.

1 THE EMBODIMENTS OF THE INVENTION FOR WHICH AN
2 EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS
3 FOLLOWS:
4

5 1. A decontamination formulation comprising:

6 (a) from about 1% to about 15% by weight of a chloroisocyanuric
7 acid;

8 (b) from about 1% to about 10% by volume of a co-solvent selected
9 from the group consisting of polypropylene glycol, polyethylene glycol, and
10 derivatives and mixtures thereof;

11 (c) from about 1% to about 15% by volume of a surfactant;

12 (d) a buffer to maintain said formulation at a pH from about 11 to
13 about 8.5; and

14 (e) the balance being water.
15

16 2. A decontamination formulation of claim 1, wherein the
17 chloroisocyanuric acid is in an amount from about 3% to about 9% by weight.
18

19 3. The decontamination formulation of claim 1, wherein said
20 chloroisocyanuric acid is selected from the group consisting of an alkali metal of
21 monochloroisocyanuric acid, dichloroisocyanuric acid, and a combination thereof
22 with cyanuric acid.
23

24 4. The decontamination formulation of claim 3, wherein said alkali
25 metal of dichloroisocyanuric acid is sodium dichloroisocyanurate.
26
27

1 5. The decontamination formulation of claim 4, wherein said buffer
2 fails over time, allowing the pH to fall to a pH about 8.5.

3

4 6. The decontamination formulation of claim 5, wherein the buffer
5 maintains the pH of the formulation above 8.5 for at least 30 minutes.

6

7 7. The decontamination formulation of claim 1, wherein the co-
8 solvent is in an amount of from about 6% to about 10% by volume.

9

10 8. The decontamination formulation of claim 1, wherein
11 polypropylene glycol has the chemical formula $R_1-(OCH(CH_3)CH_2)_n-OR_2$, where
12 R_1 and R_2 are independently H, an alkyl, or an ester group and $n > 1$.

13

14 9. The decontamination formulation of claim 8, wherein said alkyl
15 group representing R_1 or R_2 is a methyl, ethyl, propyl, or butyl group or a mixture
16 thereof.

17

18 10. The decontamination formulation of claim 8, wherein at least
19 one of said R_1 or R_2 is hydrogen.

20

21 11. The decontamination formulation of claim 8, wherein said both
22 R_1 and R_2 are hydrogens.

23

24 12. The decontamination formulation of claim 1, wherein said
25 polypropylene glycol derivative is a partially etherified polypropylene glycol.

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13. The decontamination formulation of claim 12, wherein said partially etherified polypropylene glycol has the formulae $R_1-(OCH(CH_3)CH_2)_n-OR_2$, where one of R_1 or R_2 is independently H, or an alkyl group and $n \geq 1$.

14. The decontamination formulation of claim 13, wherein said alkyl representing R_1 or R_2 is a methyl, ethyl, propyl, butyl group or a mixture thereof.

15. The decontamination formulation of claim 13, wherein at least one of said R_1 or R_2 is hydrogen.

16. The decontamination formulation of claim 1, wherein said buffer is capable of initially maintaining said formulation at a pH of from about 10 to about 11.

17. The decontamination formulation of claim 1, wherein said buffer comprises a mixture of sodium tetraborate decahydrate, anhydrous sodium carbonate and sodium hydroxide.

18. The decontamination formulation of claim 1, wherein said buffer comprises a mixture of sodium tetraborate decahydrate, anhydrous sodium carbonate and sodium metasilicate pentahydrate.

1 19. The decontamination formulation of claim 1, wherein said
2 surfactant comprises a composition of the formulae $[R(OCH_2CH_2)_nX]_aM_b$, where
3 R is an alkyl group having from eight to eighteen carbon atoms; n is an integer
4 from 0 to 10; X is selected from the group consisting of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and
5 PO_4^{3-} ; M is an alkali metal, alkaline earth metal, ammonium or amine derivatives;
6 a is the valence of M and b is the valence of $[R(OCH_2CH_2)_nX]$ or mixtures thereof.

7

8 20. The decontamination formulation of claim 1 wherein said
9 surfactant comprises a composition of the formulae $[R-CH=CH(CH_2)_m-X]_aM_b$
10 where R is an alkyl group having from eight to eighteen carbon atoms; m is an
11 integer from 0 to 3; X is selected from the group of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and PO_4^{3-} ,
12 M is an alkali metal, alkaline earth metal, ammonium or amine derivatives; a is
13 the valence of M and b is the valence of $[R-CH=CH(CH_2)_m-X]$ or mixtures thereof.

14

15 21. The decontamination formulation of claim 1 wherein said
16 surfactant comprises a composition of the formula $R-OH$, where R is an alkyl
17 group having from eight to sixteen carbon atoms or mixtures thereof.

18

19 22. The decontamination formulation of claim 1 wherein said
20 surfactant comprises a composition as described in claims 7, 8, 9, 10 and 11.

21

22 23. The decontamination formulation of claim 1, further comprising
23 lithium hypochlorite in an amount of from about 5% to about 10% by weight of
24 said chloroisocyanuric acid salt.

25

1 24.A method of preparing a decontamination formulation
2 comprising the steps of adding to a stream of water:

3 (a) a first aqueous solution comprising of up to about 30% by weight
4 of chloroisocyanuric acid;

5 (b) a second aqueous solution comprising a mixture of inorganic
6 buffer salts adjusted to an initial pH of about 10 to 11 and capable of maintaining
7 the pH of said decontamination formulation from about 11 to about 8.5;

8 (c) a co-solvent selected from the group consisting of polypropylene
9 glycol, polyethylene glycol and a derivative and mixture thereof; and

10 (d) a surfactant.

11

12 25.The method of claim 24, wherein the surfactant is a foaming
13 agent.

14

15 26.The method of claim 24, wherein the surfactant is a foaming
16 agent comprising a composition of the formula $[R(OCH_2CH_2)_nX]_aM_b$, where R is
17 an alkyl group having from eight to eighteen carbon atoms; n is an integer from 0
18 to 10; X is selected from the group of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and PO_4^{3-} ; M is an alkali
19 metal, alkaline earth metal, ammonium or amine derivatives; a is the valence of
20 M and b is the valence of $[R(OCH_2CH_2)_nX]$.

21

22 27.The method of claim 22, wherein the surfactant is a foaming
23 agent comprising a composition of the formula $[R-CH=CH(CH_2)_m-X]_aM_b$ where R
24 is an alkyl group having from eight to eighteen carbon atoms; m is an integer
25 from 0 to 3; X is selected from the group of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and PO_4^{3-} , M is an

1 alkali metal, alkaline earth metal, ammonium or amine derivatives; a is the
2 valence of M and b is the valence of $[R-CH=CH(CH_2)_m-X]$.

3

4 28. The method of claim 24 wherein said surfactant comprises a
5 composition of the formula R-OH, where R is an alkyl group having from eight to
6 sixteen carbon atoms or mixtures thereof.

7

8 29. The method of claim 24 wherein said surfactant comprises a
9 composition as described in claims 7,8,9,10 and 11.

10

11 30. The method of claim 24, wherein said first aqueous solution
12 additionally comprises a lithium hypochlorite in amounts of up to 10% of the
13 chloroisocyanuric acid salt.

14

15 31. A kit for providing a decontamination composition comprising the
16 following components in packaged form:

17 (a) a decontaminant comprising chloroisocyanuric acid; or its alkali
18 metal or alkaline earth metal salt or a substance thereof;

19 (b) a co-solvent selected from the group consisting of polypropylene
20 glycols, polyethylene glycols, and derivatives and mixtures thereof;

21 (c) a surfactant; and

22 (d) a mixture of sodium tetraborate decahydrate, anhydrous sodium
23 carbonate and sodium hydroxide.

24

1 32. A kit as claimed in claim 31, wherein said decontaminant further
2 includes lithium hypochlorite.

3

4 33. A kit as claimed in claim 31, wherein said chloroisocyanuric acid
5 is sodium dichloroisocyanurate.

6

7 34. A kit as claimed in claim 31, wherein said surfactant comprises a
8 composition of the formulae $[R(OCH_2CH_2)_nX]_aM_b$, where R is an alkyl group
9 having from eight to eighteen carbon atoms; n is an integer from 0 to 10; X is
10 selected from the group of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and PO_4^{3-} ; M is an alkali metal,
11 alkaline earth metal, ammonium or amine derivatives; a is the valence of M and b
12 is the valence of $[R(OCH_2CH_2)_nX]$ or a mixture thereof.

13

14 35. A kit as claimed in claim 31, wherein said surfactant comprises a
15 composition of the formulae $[R-CH=CH(CH_2)_m-X]_aM_b$ where R is an alkyl group
16 having from eight to eighteen carbon atoms; m is an integer from 0 to 3; X is
17 selected from the group of SO_3^{2-} , SO_4^{2-} , CO_3^{2-} and PO_4^{3-} ; M is an alkali metal,
18 alkaline earth metal, ammonium or amine derivatives; a is the valence of M and b
19 is the valence of $[R-CH=CH(CH_2)_m-X]$.

20

21 36. A kit as claimed in claim 31 wherein said surfactant comprises a
22 composition of the formula $R-OH$, where R is an alkyl group having from eight to
23 sixteen carbon atoms or mixtures thereof.

1 37. A kit as claimed in claim 31 wherein said surfactant comprises a
2 composition as described in claims 7,8,9,10 and 11.

3

4 38. A kit as claimed in claim 31, wherein said composition
5 components (a) and (b) are individually packaged and components (c) and (d)
6 are packaged as a mixture or components (a) and (d) are packaged as a mixture
7 and components (b) and (c) are packaged as a mixture.

8

9 39. A kit as claimed in claim 31, wherein said composition
10 components are individually packaged.

11

12 40. A method for decontaminating surfaces comprising the steps of:

13 (a) preparing a decontamination formulation of from about 1% to
14 about 15% by weight of a chloroisocyanuric acid salt, from about 1% to about
15 10% by volume of a co-solvent selected from the group consisting of
16 polypropylene glycol, polyethylene glycol, and derivatives and mixtures thereof,
17 from about 1% to about 15% by volume of a surfactant, and a buffer to initially
18 maintain said formulation at a pH from about 11 to about 8.5 and water to form an
19 aqueous solution; and

20 (b) applying the aqueous solution to contaminated surfaces.

21

22 41. The decontamination method of claim 40, wherein said buffer
23 fails over time, allowing the pH to fall to a pH about 8.5.

24

1 42. The decontamination method of claim 41 wherein the buffer
2 maintains the pH of the aqueous solution above 8.5 for at least 30 minutes.

3

4 43. The decontamination method of claim 40 further comprising the
5 steps of:

6 (a) foaming the aqueous solution; then

7 (b) applying the foamed aqueous solution to the contaminated
8 surface.

9

10 44. The decontamination method of claim 41 wherein the foaming
11 step comprises dispensing the aqueous solution through an aeration nozzle.

12

13 45. The decontamination method of claim 44, wherein said buffer
14 fails over time, allowing the pH to fall to a pH about 8.5.

15

16 46. The decontamination method of claim 45 wherein the buffer
17 maintains the pH of the aqueous solution above 8.5 for at least 30 minutes.

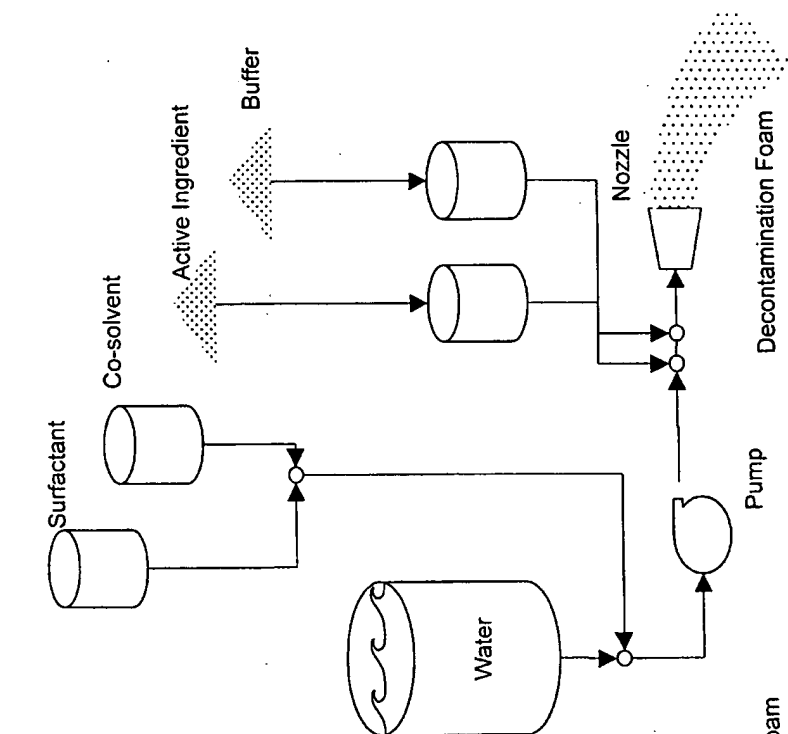
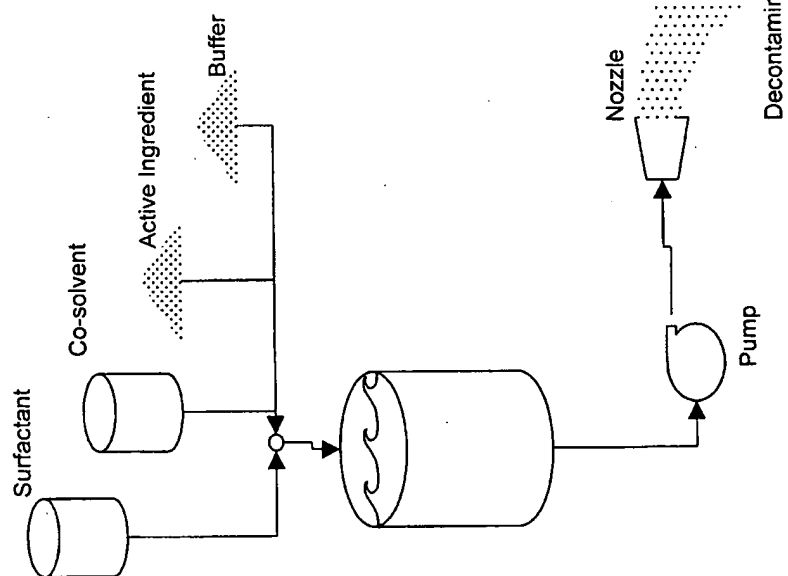
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19 47. The decontamination method as recited in claim 40 wherein all
20 of the chloroisocyanuric acid salt, co-solvent, surfactant, and the buffer are
21 combined with water before applying to the contaminated surface.

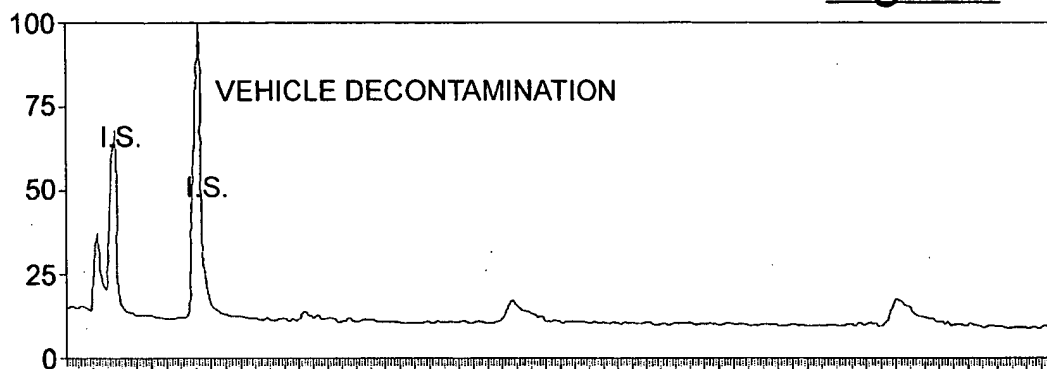
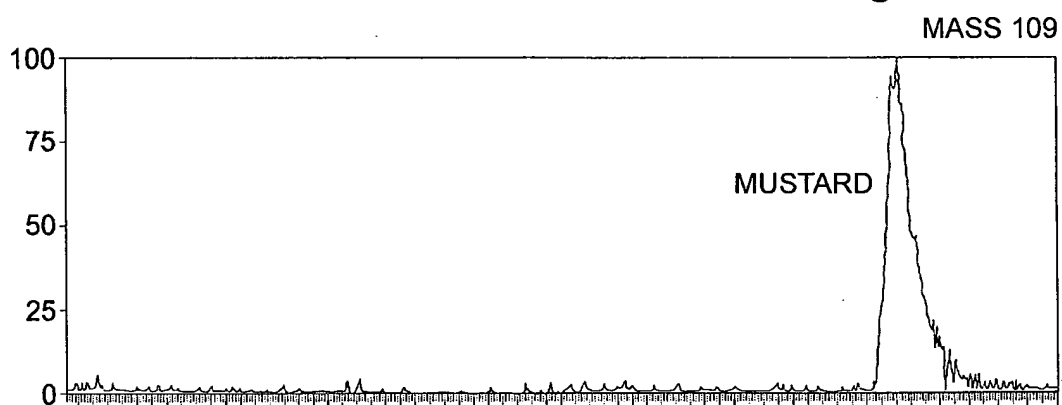
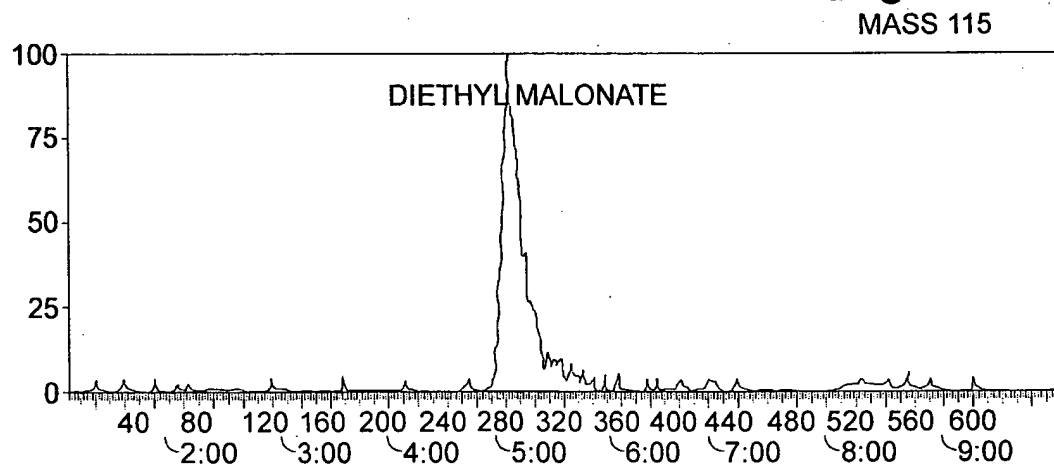
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1 48. The decontamination method as recited in claim 40 wherein
2 (a) the co-solvent and surfactant are combined with water to form a
3 non-degrading solution; and
4 (b) the buffer and the chloroisocyanuric acid salt are added
5 separately to the non-degrading solution before applying to the contaminated
6 surface.
7
8

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Fig. 1b**Fig. 1a**

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Fig. 2aFig. 2bFig. 2c

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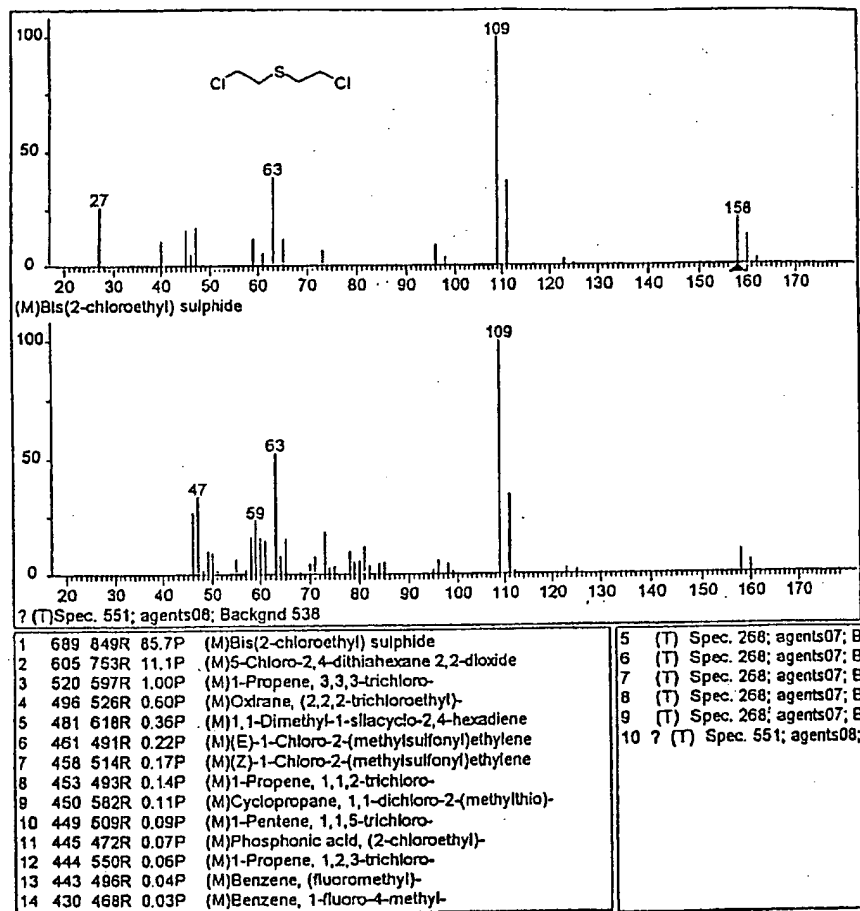
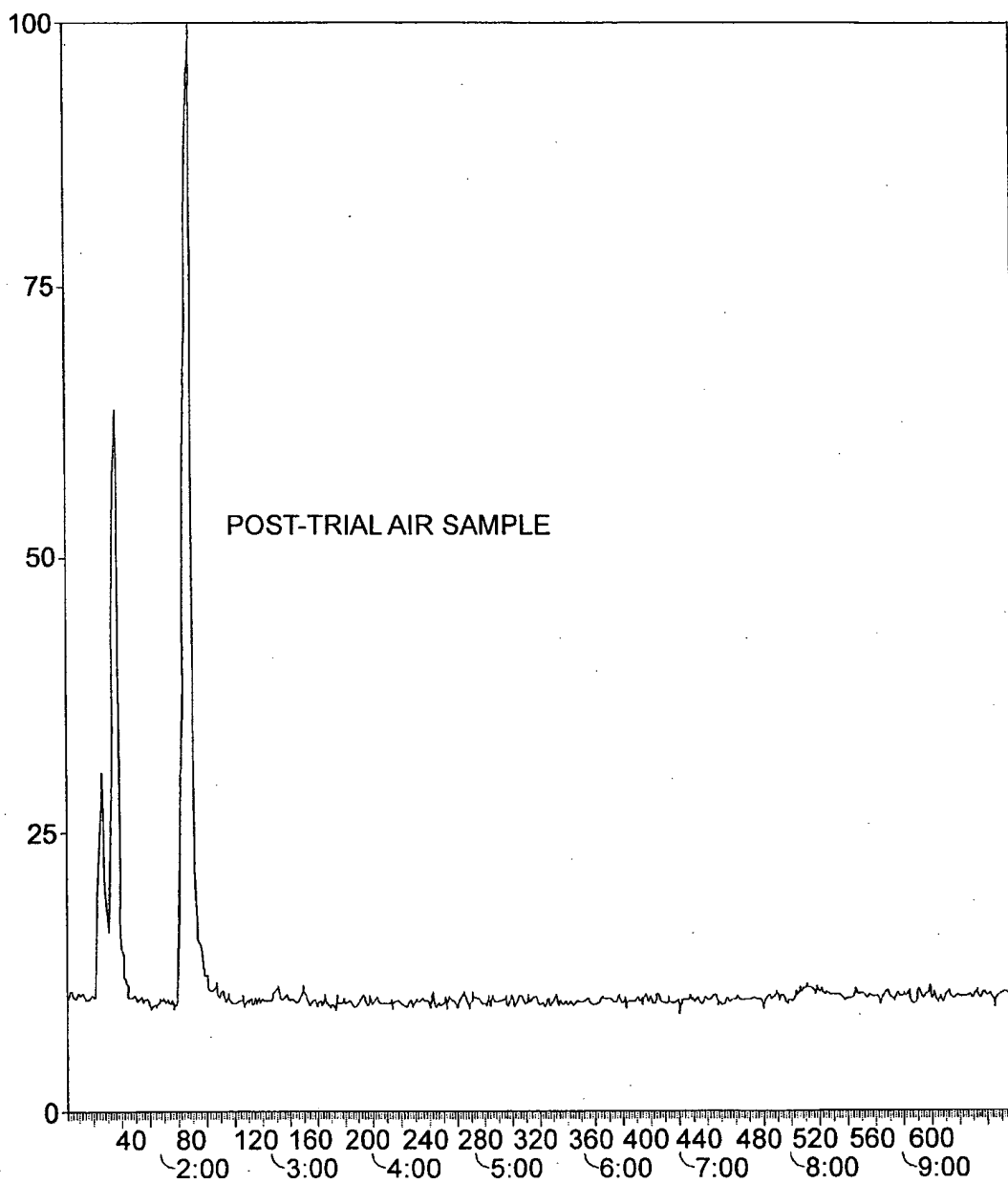


Fig. 3

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Fig. 4

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Fig. 5

Agent Under Test	w/w% Agent	w/w% SD	SD/ Agent	Buffer Strength	w/w% Foamer/ Surfactant	w/w% Cosolvent	Time (Min)	% Agent Remaining		LC-MS Ions Monitored
								LC-FPD	LC-MS	
GB	0.29	3.0	5.7	0.67	1.3	0	0.43	<0.56	-	-
							3.7	-	<0.2	99.1, 141.1
							5.6	<0.56	-	-
							10.8	-	<0.2	99.1, 141.1
							56.6	<0.56	-	-
GB	0.29	6.0	11.4	1.0	4.6	6.9	1.13	<0.56	-	-
							3.52	-	<0.2	99.1, 141.1
							4.92	<0.56	-	-
							10.0	-	<0.2	99.1, 141.1
GB	0.29	9.0	17.2	1.0	4.8	6.9	1.78	<0.56	-	-
							5.43	<0.56	-	-
GA	0.29	3.0	5.7	0.67	1.6	0	1.33	0	-	-
							2.43	0	-	-
							4.95	0	-	-
							53.0	0	-	-
							21.8hrs	0	-	-
GA	0.29	3.0	5.7	0.67	1.8	0	1.07	0	-	-
							3.43	-	<0.46	163.1
							4.85	0	-	-
							9.67	-	<0.46	163.1
GA	0.29	6.0	11.4	1.0	4.5	7.5	1.07	0	-	-
							3.35	-	<0.46	163.1
							4.85	0	-	-
							10.0	-	<0.46	163.1
							360	0	-	-
GD	0.29	3.0	5.7	0.67	1.6	0	1.07	5.0	-	-
							2.17	tr	-	-
							3.53	tr	tr	183.1
							4.77	0.5	-	-
							10.0	-	<0.06	183.1
GD	0.29	6.0	11.4	1.0	4.6	7.8	1.08	4.3	-	-
							2.22	tr	-	-
							3.53	tr	tr	183.1
							4.85	1.4	-	-
							9.98	-	<0.06	183.1
HD	0.27	3.0	5.7	0.67	1.6	0.0	2.67	0.0	-	-
							4.92	0.0	-	-
HD	0.27	3.0	5.7	0.67	1.6	0.0	3.0	0.0	-	-
							5.4	6.2	-	-
							62.1	0.0	-	-
HD	0.27	3.0	5.7	0.67	1.6	0.0	3.65	0.0	-	-
							4.97	0.0	-	-
HD	0.27	6.0	11.4	1.0	4.7	7.2	2.47	0.0	-	-
							5.27	0.0	-	-
							53.3	0.0	-	-

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Fig. 6

Formulation	uL/mL	VX Molar ratio SD/NX	mg weighed	Reaction Time (minutes)	Residual VX	Product Toxic ?	Notes
Buffer/Surfactant	4			6 days	42%	YES	
FS* w/SD	12			125	>50%	YES	VX droplets not solubilizing
FS* w/SD	12			6 days	>50%	YES	
FS*	12	18.2		24	0	0	
FS*	12	18.2		7	0	0	
Mild	12	6.3	11.6	6	24.90%	NO	
Mild	12	6.0	12.2	11	20.90%	Trace	
Mild	12	6.0	12.2	15	11.40%	Trace	
Mild	12	6.2	11.7	33	11.80%	Trace	
Mild	12	6.3	11.6	230	19.40%	Trace	
Mild	12	6.3	11.6	3 days 23 hrs	50.10%	Trace	
Mild	12	6.3	11.6	5 days	28.77%		
Mild	4	16.9	4.3	7	1.28%	Trace	
Mild	4	18.2	4	11	0.63%	Trace	
Mild	4	18.2	4	66	0.65%	Trace	
Mild	4	18.2	4	189	0.56%	Trace	
Mild	3	29.1	2.5	12	0.68%	Trace	
Mild	0	0	0	-	0	-	

* Full 9% SD Strength

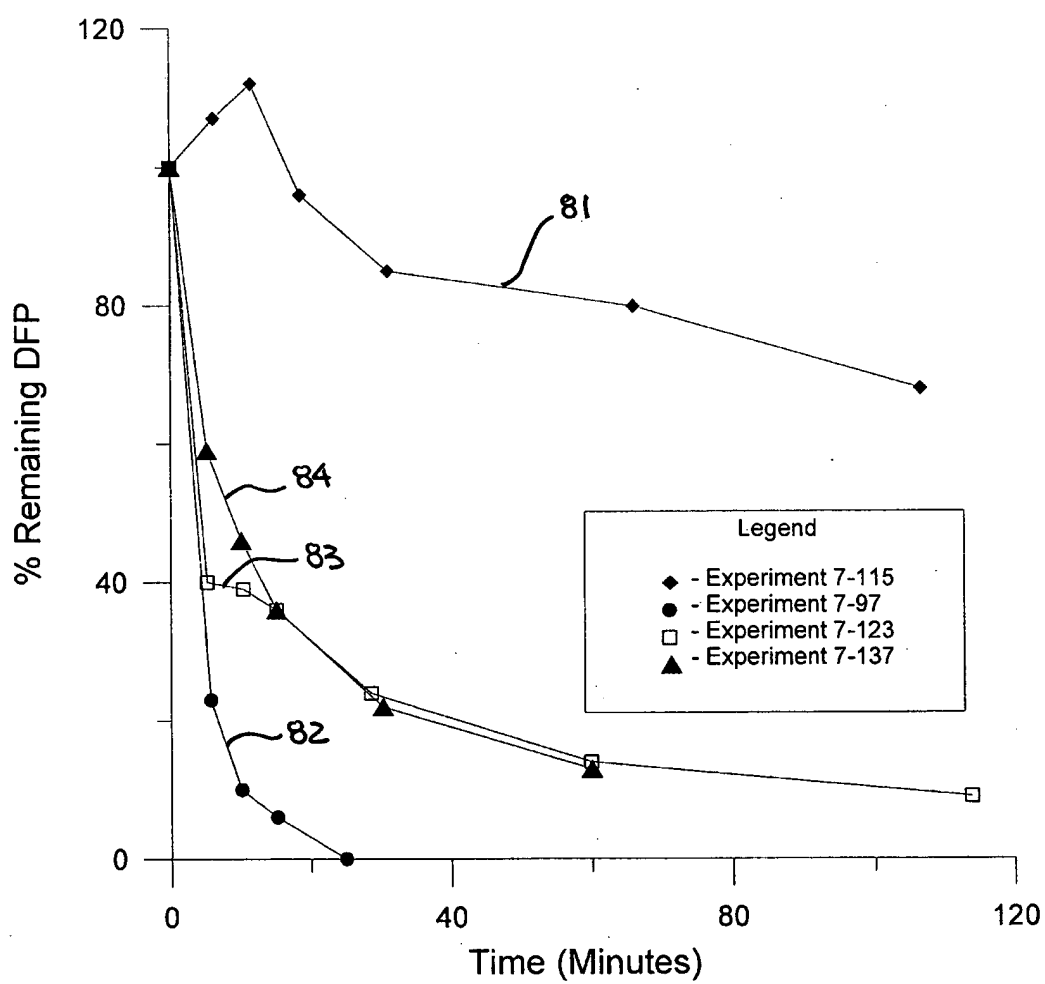
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Fig. 7

	Test	% DFP	% SD	% LiOCl	% Foamer	% Cosolvent	Time	% DFP	% DFP
		(w/w)	(w/w)	(w/w)	(w/w)	(w/w)	(Min)	Residual	at 30 min.
SD	7-115	1.24	0	0	0	0	0.00	100	87
							6.25	107	
							11.58	112	
							18.60	96	
							31.25	85	
							66.00	80	
							106.77	68	
	7-97	1.25	7.33	0	0	0	0.00	100	3
							5.67	23	
							10.05	10	
							15.17	6	
							25.05	0	
	7-123	1.34	7.97	0	0	7.2	0.00	100	25
							5.17	40	
							10.30	39	
							15.05	36	
							28.72	24	
							59.85	14	
	7-137	1.25	7.42	0	3.5	7.6	0.00	100	21
							5.13	59	
							10.05	46	
							15.10	36	
							30.43	22	
							60.00	13	
SD+KBr	7-143	0.26	13.72	0	3.4	6.3	0.00	100	23
							5.30	61	
							9.88	43	
							14.75	32	
							29.82	23	
							60.02	16	
							118.00	11	
	7-147	0.36	15	0	3.5	8.5	0.00	100	8
							5.27	12	
							10.07	26	
							15.63	20	
							30.43	8	
							59.67	6	
SD + LiOCl	8-21	0.29	11.32	0	1.5	0	0.00	100	2
							4.98	40	
							10.00	20	
							15.08	10	
							30.00	2	
							60.38	0	
							120.00	0	
	8-23	0.32	6.52	0.19	1.5	0	0.00	100	0.5
							4.88	17	
							10.05	6	
							15.03	3	
							30.00	1	
							60.00	0	
							120.00	0	

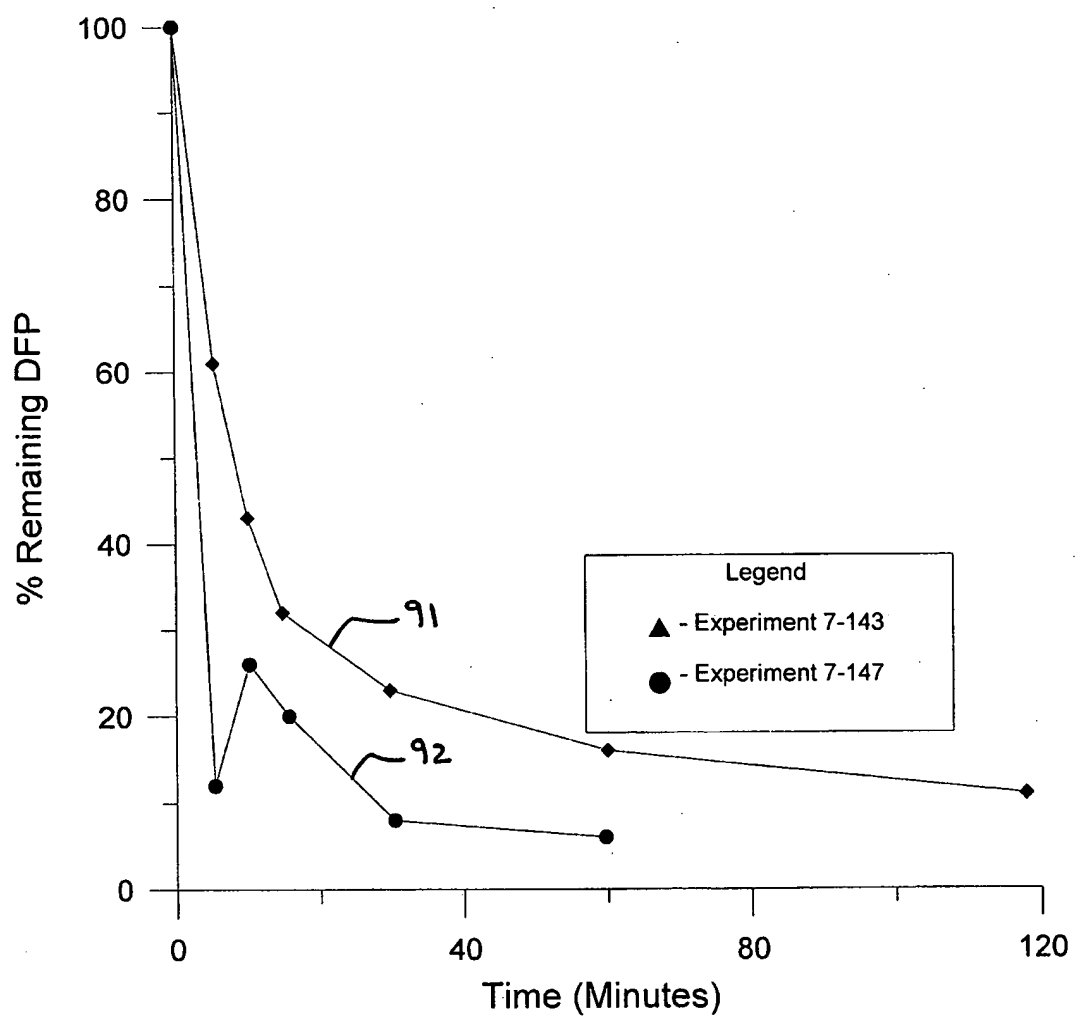
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Fig. 8



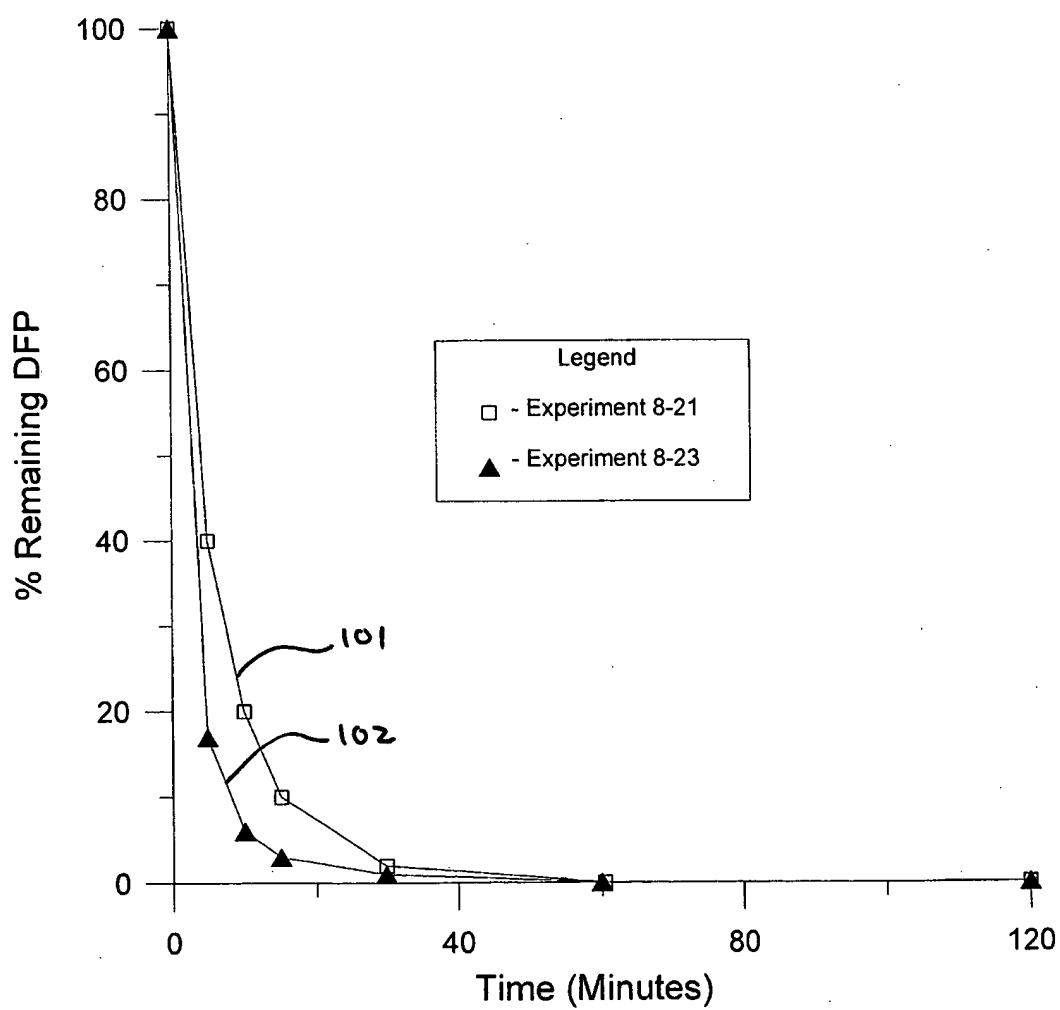
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Fig. 9

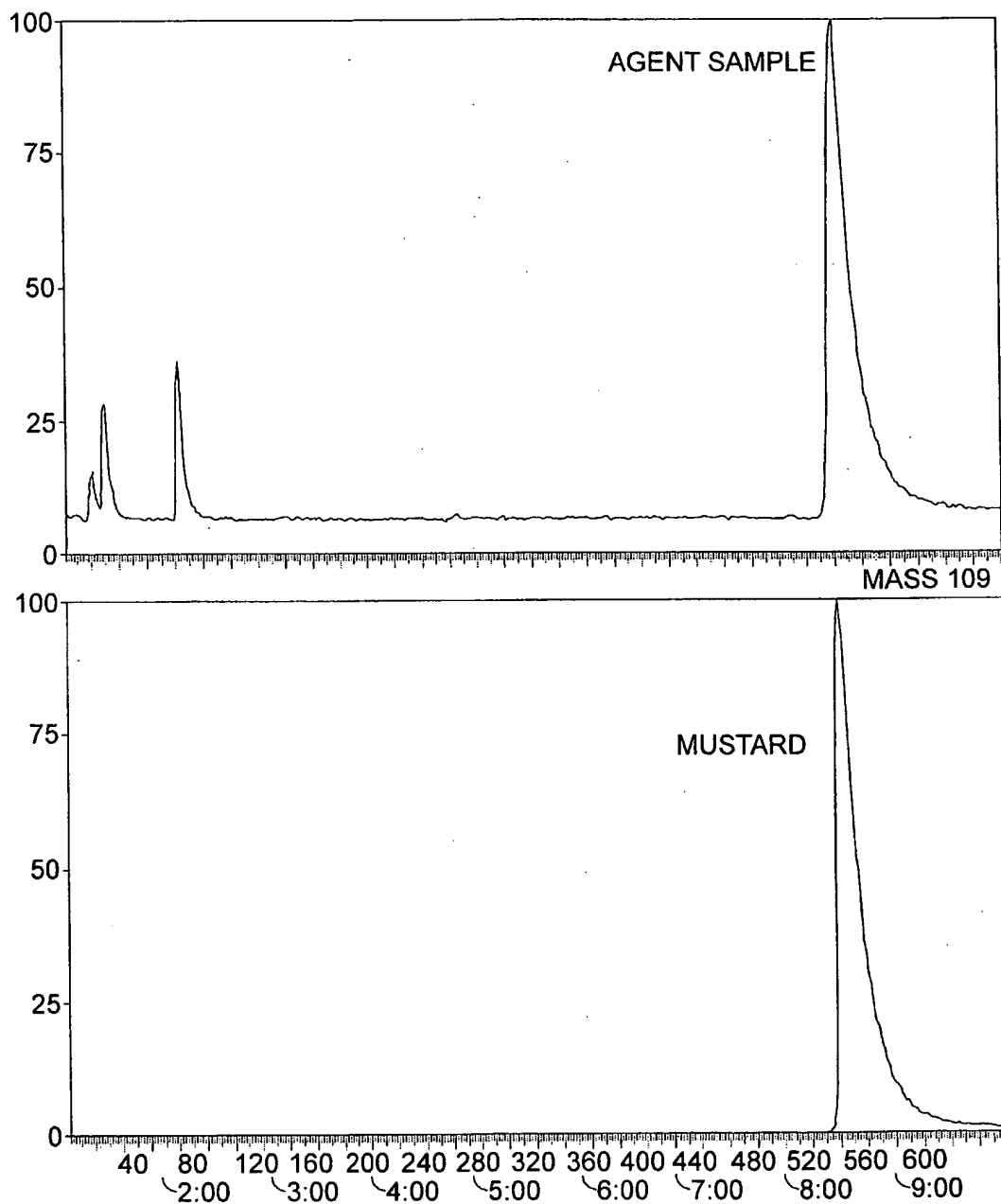


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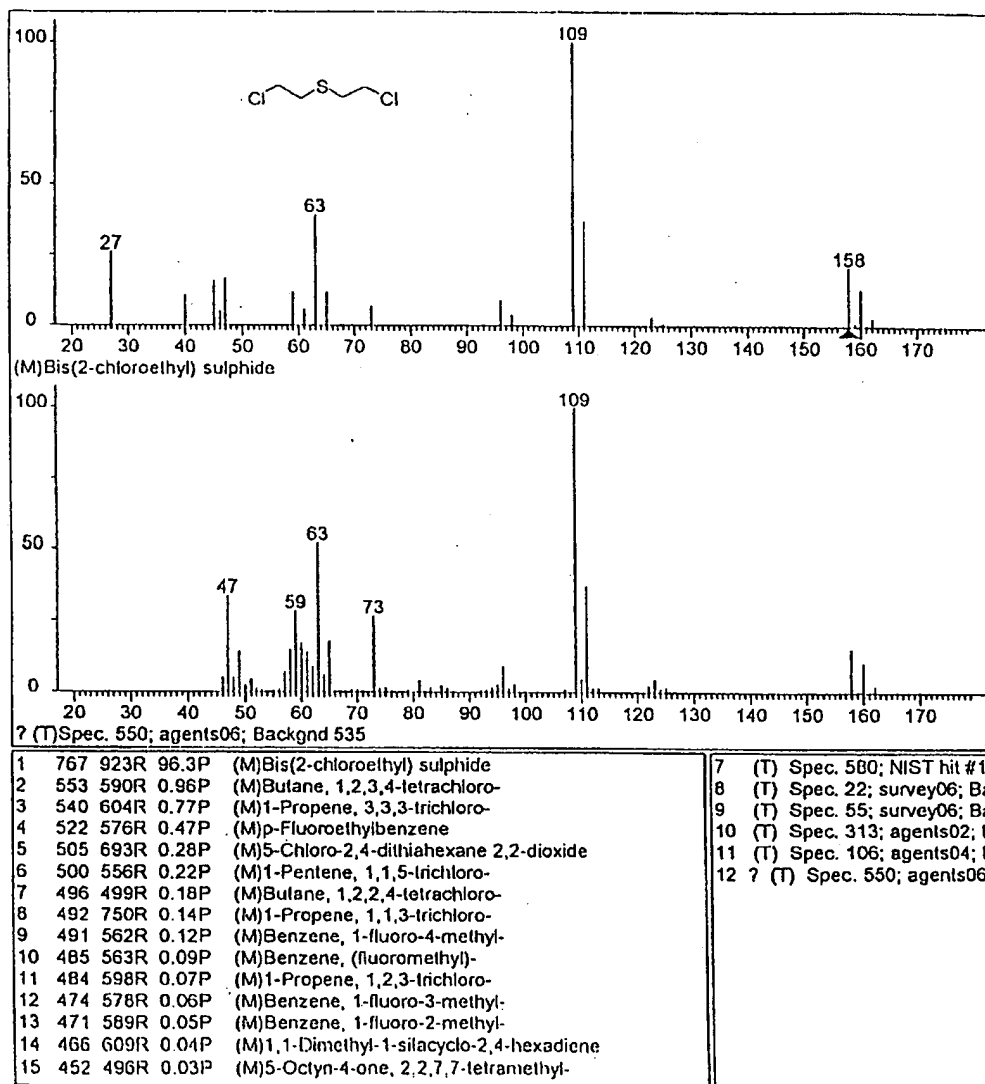
Fig. 10



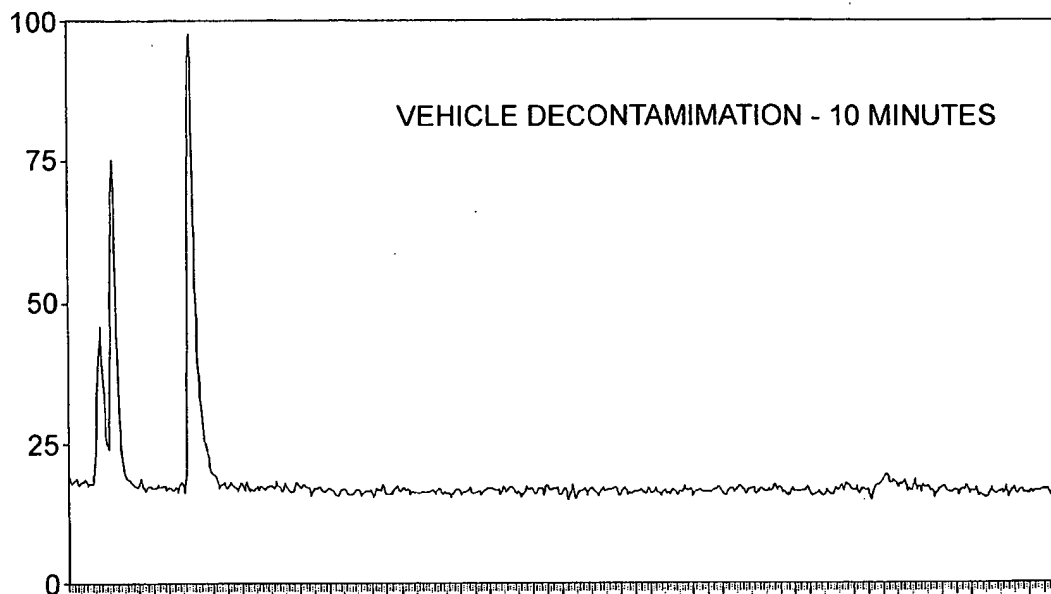
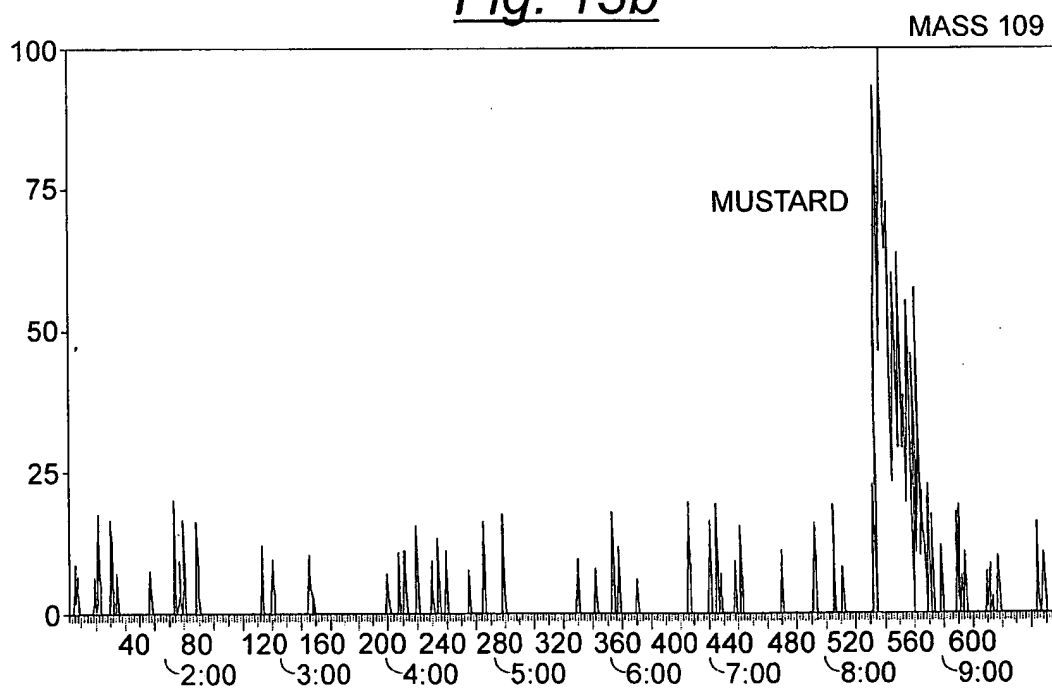
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Fig. 11

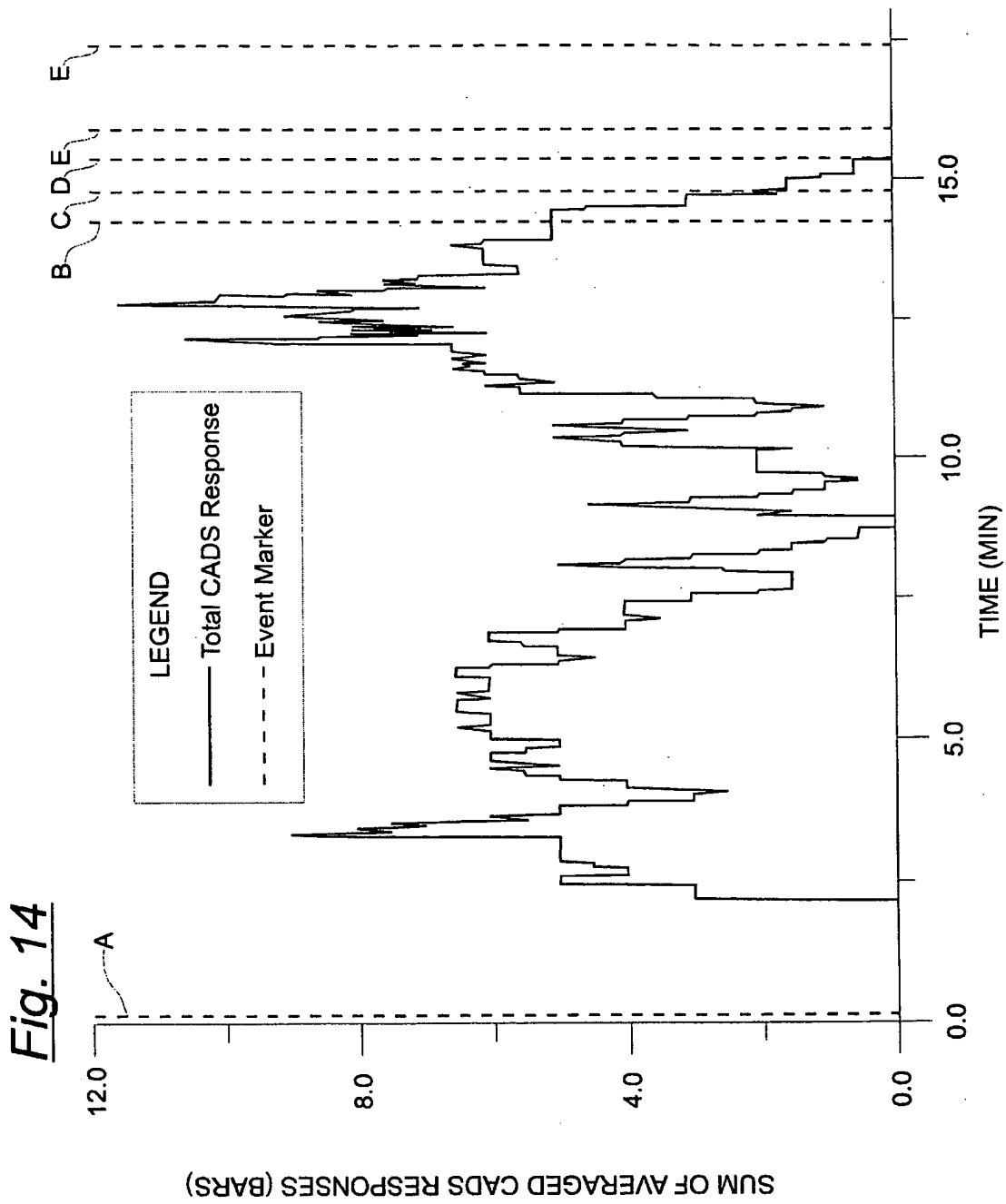
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Fig. 12

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Fig. 13aFig. 13b

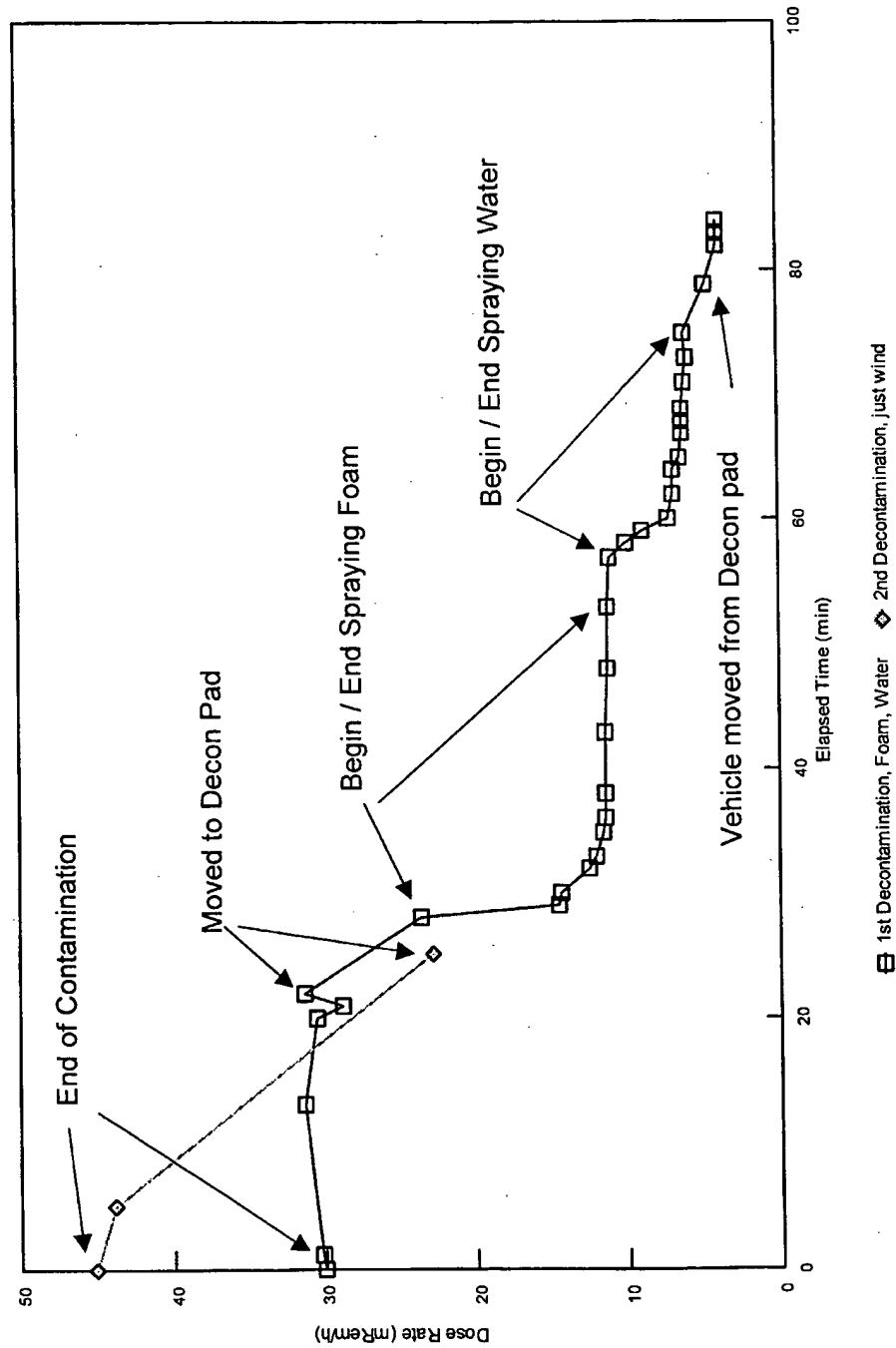
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Nuclear Particle Decontamination

Fig. 15



INTERNATIONAL SEARCH REPORT

International Application No

PCT/CA 00/00137

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A62D3/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A62D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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P,X	PATENT ABSTRACTS OF JAPAN vol. 1999, no. 11, 30 September 1999 (1999-09-30) & JP 11 148098 A (KAO CORP), 2 June 1999 (1999-06-02) abstract --- -/--	1-3,7, 24,25, 40,41, 43,47,48



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